Degussa

Degussa
Corporation

PDCN: 88940000224

8EHQ-0495-12982

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95 APR -4 PM 12:02

ORIGINAL

March 29, 1995

Document Processing Center (7407)
Attn: TSCA Section 8(e) Coordinator
Office of Pollution Prevention and Toxics
Environmental Protection Agency
401 M Street, S. W.
Washington, D.C. 20460-0001

(B)

8EHQ-94-12982
SP001 04/04/95

Re: Response to EPA Information Request
Substantial Risk Notification Section 8(e)
8EHQ-94-12982

Contains no CBI

Dear Sir:

This submission is a response to Terry O'Bryan's request for information regarding Substantial Risk Notification 8EHQ-94-12982. The chemical substance is Methanesulfinic acid, aminoimino- (which we call Formamidine Sulfinic Acid or "FAS"), CASRN 1758-73-2. We import this chemical substance.

EPA's questions are addressed below, and in the attachments.

1. Additional information of commercial uses and any other information to assist EPA in assessing exposure potential.

We are not aware of commercial or consumer uses. The major uses are bleaching and decolorizing waste paper and textiles. These are considered industrial uses

The potential exposure to FAS is limited to the workplace. Worker exposure is minimized by exposure controls which are described in the MSDS. In most of the applications, about 6 pounds of FAS are utilized for each ton of pulp (100% basis). In the process FAS is completely consumed and forms principally urea and sodium bisulfate. (The reaction equation is shown in Figure 1 in Attachment 2).

USES

A. Bleaching waste paper for recycling. Descriptions are detailed in a brochure entitled "Application in Paper Recycling / FAS" (Attachment 1) and in a paper entitled "Advanced Bleaching Technology for Secondary Fibers" (Attachment 2).

B. Decolorizing waste paper.

C. Bleaching textiles.

D. Decolorizing textile effluent.

E. Free radical initiator in some polymerization reactions.

89950000167

2. A description of all voluntary actions taken by us in response to the findings indicated in our submission.

A. Degussa Corporation's voluntary actions are described in a memo from our Manager of Corporate Industrial Hygiene (Attachment 3) which was the basis of a memo sent to our sales force (Attachment 4).

RECEIVED mn 4/12/95

65 Challenger Road Ridgefield Park, NJ 07660 (201) 641-6100

B. The sales force personally notified all of our FAS customers. In addition, letters containing information about the mutagenicity test results and in some cases, safety information, were sent to those customers who requested letters for their own files.

C. We modified the FAS Material Safety Data Sheet (Attachment 5) to put a stronger emphasis on ventilation controls to eliminate airborne dust at the source point, and to emphasize the avoidance and minimization of dust formation.

D. If a positive mutagenicity result is published, we intend to add the information to the MSDS.

E. According to our German parent company, Degussa AG, several other mutagenicity studies have been performed with FAS. In an Ames test, FAS was weakly positive. In two mammalian cell gene mutation tests (HGPRT locus test, Chinese hamster cells) negative results were obtained.

F. Three in-vivo micronucleus tests in mice have been performed. In an older Degussa test with oral administration, the test results are equivocal.

Therefore in the OECD SIDS Review Meeting in Paris (September 20-23, 1993) it was decided to perform an additional test in Austria (Austria is the contact point for FAS). Unfortunately, evaluation of the results of this micronucleus test in mice with oral administration was ambiguous.

Degussa AG hired a consultant toxicologist to review the Austrian mutagenicity study.

G. The German authorities, BGV V (formerly called BGA) do not consider FAS to be a mutagen. They have reviewed the report of Degussa's consultant. They suggested that the test be repeated because the results were not suitable for making a classification. The test will be repeated at an independent laboratory.

H. Degussa AG informed their customers about the issue of FAS genotoxicity. One of the customers furnished the results of a micronucleus test with interperitoneal administration which showed negative results.

Please let me know if you want additional information.

Sincerely,

John Lewinson

John Lewinson, Ph.D.
Manager, Product Regulatory Compliance

JL-95-142

cc: M. Berger, DCRP
Dr. Kronis, ATC
R. Marion, DCA
R. Wardell, DCRP

Triage of 8(e) Submissions

Date sent to triage: JAN 11 19[illegible]

(NON-CAP) CAP

Submission number: 12982 A

TSCA Inventory: (Y) N D

Study type (circle appropriate):

Group 1 - Dick Clements (1 copy total)

ECO **AQUATO**

Group 2 - Ernie Falke (1 copy total)

ATOX SBTOX SEN w/NEUR

Group 3 - Elizabeth Margosches (1 copy each)

STOX CTOX EPI RTOX (GTOX)

STOX/ONCO CTOX/ONCO IMMUNO CYTO NEUR

Other (FATE, EXPO, MET, etc.): ____________________

Notes:

THIS IS THE ORIGINAL 8(e) SUBMISSION; PLEASE REFILE AFTER TRIAGE DATABASE ENTRY

For Contractor Use Only

entire document: (0) 1 2 **pages** 1 **pages** 1

Notes:

Contractor reviewer : NEB **Date:** 11/14/94

CECATS\TRIAGE TRACKING DBASE ENTRY FORM

CECATS DATA:
Submission # 8EHQ-0994-12982 SEQ. A

TYPE: INT SUPP FLWP

SUBMITTER NAME: Degussa Corporation

INFORMATION REQUESTED: FLWP DATE:
0501 NO INFO REQUESTED
0502 INFO REQUESTED (TECH)
0503 INFO REQUESTED (VOL ACTIONS)
0504 INFO REQUESTED (REPORTING RATIONALE)

DISPOSITION:
0639 REFER TO CHEMICAL SCREENING
0678 CAP NOTICE

VOLUNTARY ACTIONS:
0401 NO ACTION REPORTED
0402 STUDIES PLANNED/UNDERWAY
0403 NOTIFICATION OF WORKER/OTHERS
0404 LABEL/MSDS CHANGES
0405 PROCESS/HANDLING CHANGES
0406 APP./USE DISCONTINUED
0407 PRODUCTION DISCONTINUED
0408 CONFIDENTIAL

SUB. DATE: 09/01/94 OTS DATE: 09/07/94 CSRAD DATE: 10/11/94

CHEMICAL NAME:

CAS# 1758-73-2

INFORMATION TYPE:		P F C	INFORMATION TYPE:		P F C	INFORMATION TYPE:		P F C
0201	ONCO (HUMAN)	01 02 04	0216	EPI/CLIN	01 02 04	0241	IMMUNO (ANIMAL)	01 02 04
0202	ONCO (ANIMAL)	01 02 04	0217	HUMAN EXPOS (PROD CONTAM)	01 02 04	0242	IMMUNO (HUMAN)	01 02 04
0203	CELL TRANS (IN VITRO)	01 02 04	0218	HUMAN EXPOS (ACCIDENTAL)	01 02 04	0243	CHEM/PHYS PROP	01 02 04
0204	MUTA (IN VITRO)	01 02 04	0219	HUMAN EXPOS (MONITORING)	01 02 04	0244	CLASTO (IN VITRO)	01 02 04
0205	MUTA (IN VIVO)	01 02 04	0220	ECO/AQUA TOX	01 02 04	0245	CLASTO (ANIMAL)	01 02 04
0206	REPRO/TERATO (HUMAN)	01 02 04	0221	ENV. OCCC/REL/FATE	01 02 04	0246	CLASTO (HUMAN)	01 02 04
0207	REPRO/TERATO (ANIMAL)	01 02 04	0222	EMER INCI OF ENV CONTAM	01 02 04	0247	DNA DAM/REPAIR	01 02 04
0208	NEURO (HUMAN)	01 02 04	0223	RESPONSE REQEST DELAY	01 02 04	0248	PROD/USE/PROC	01 02 04
0209	NEURO (ANIMAL)	01 02 04	0224	PROD/COMP/CHEM ID	01 02 04	0251	MSDS	01 02 04
0210	ACUTE TOX. (HUMAN)	01 02 04	0225	REPORTING RATIONALE	01 02 04	0299	OTHER	01 02 04
0211	CHR. TOX. (HUMAN)	01 02 04	0226	CONFIDENTIAL	01 02 04			
0212	ACUTE TOX. (ANIMAL)	01 02 04	0227	ALLERG (HUMAN)	01 02 04			
0213	SUB ACUTE TOX (ANIMAL)	01 02 04	0228	ALLERG (ANIMAL)	01 02 04			
0214	SUB CHRONIC TOX (ANIMAL)	01 02 04	0239	METAB/PHARMACO (ANIMAL)	01 02 04			
0215	CHRONIC TOX (ANIMAL)	01 02 04	0240	METAB/PHARMACO (HUMAN)	01 02 04			

TRIAGE DATA:	NON-CBI INVENTORY	ONGOING REVIEW	SPECIES	TOXICOLOGICAL CONCERN:	USE:	PRODUCTION:
	YES	YES (DROP/REFER)	mus	LOW		import
CAS SR	NO	NO (CONTINUE)		MED		
	DETERMINE	REFER:		HIGH		

COMMENTS: Non-Cap

4) 8EHQ-09954-12982: Rank - medium.

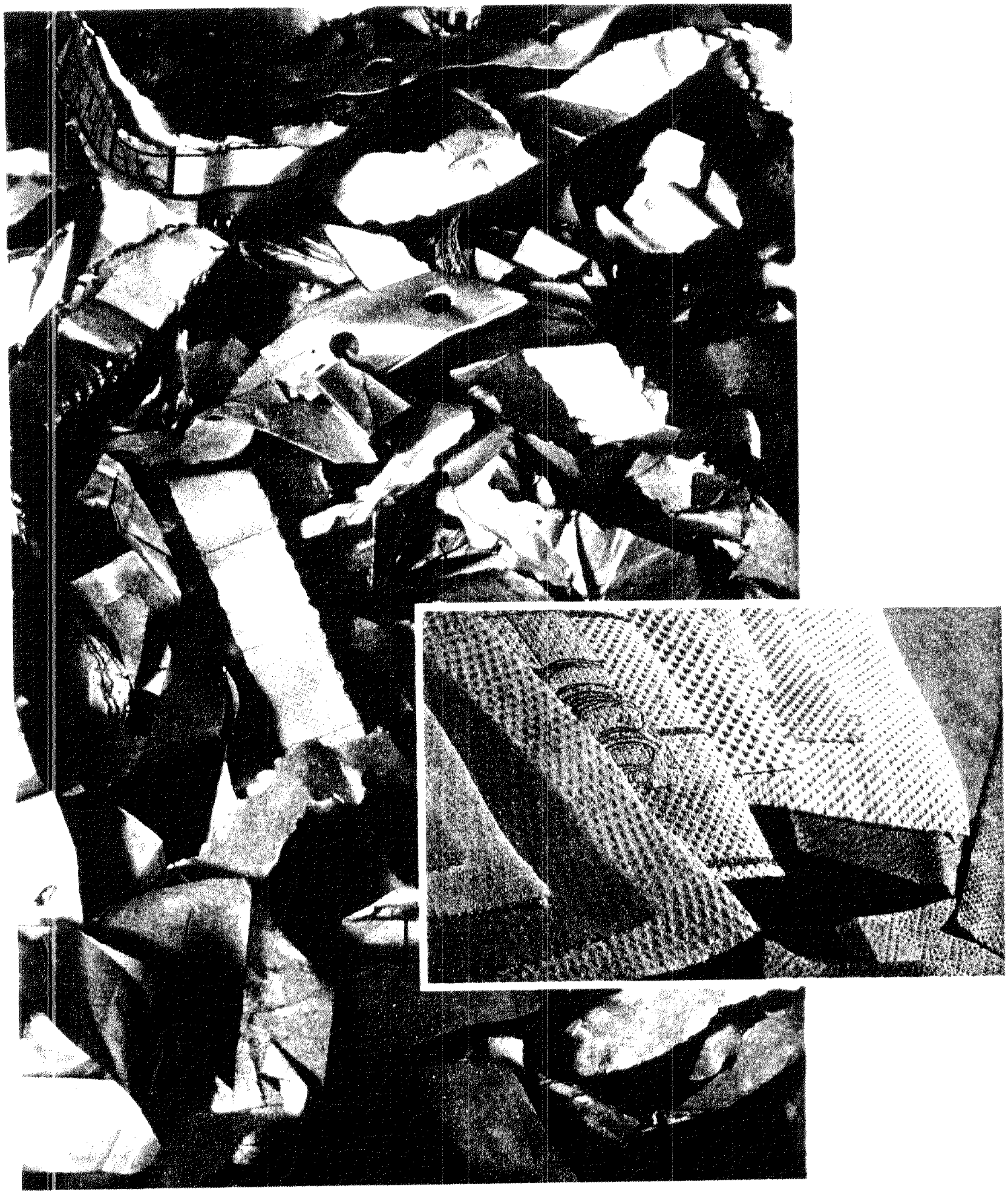
Chemical: aminoiminomethanesulfinic acid (formamidine sulfinic acid; FAS; CAS# 1758-73-2)

Letter from Degussa Corp, Ridgefield Park NJ, dated September 1, 1994: Positive for chromosome mutations (micronuclei) in mice exposed in vivo by oral gavage.

Application in Paper Recycling

FAS (Formamidine sulfinic acid)

Degussa



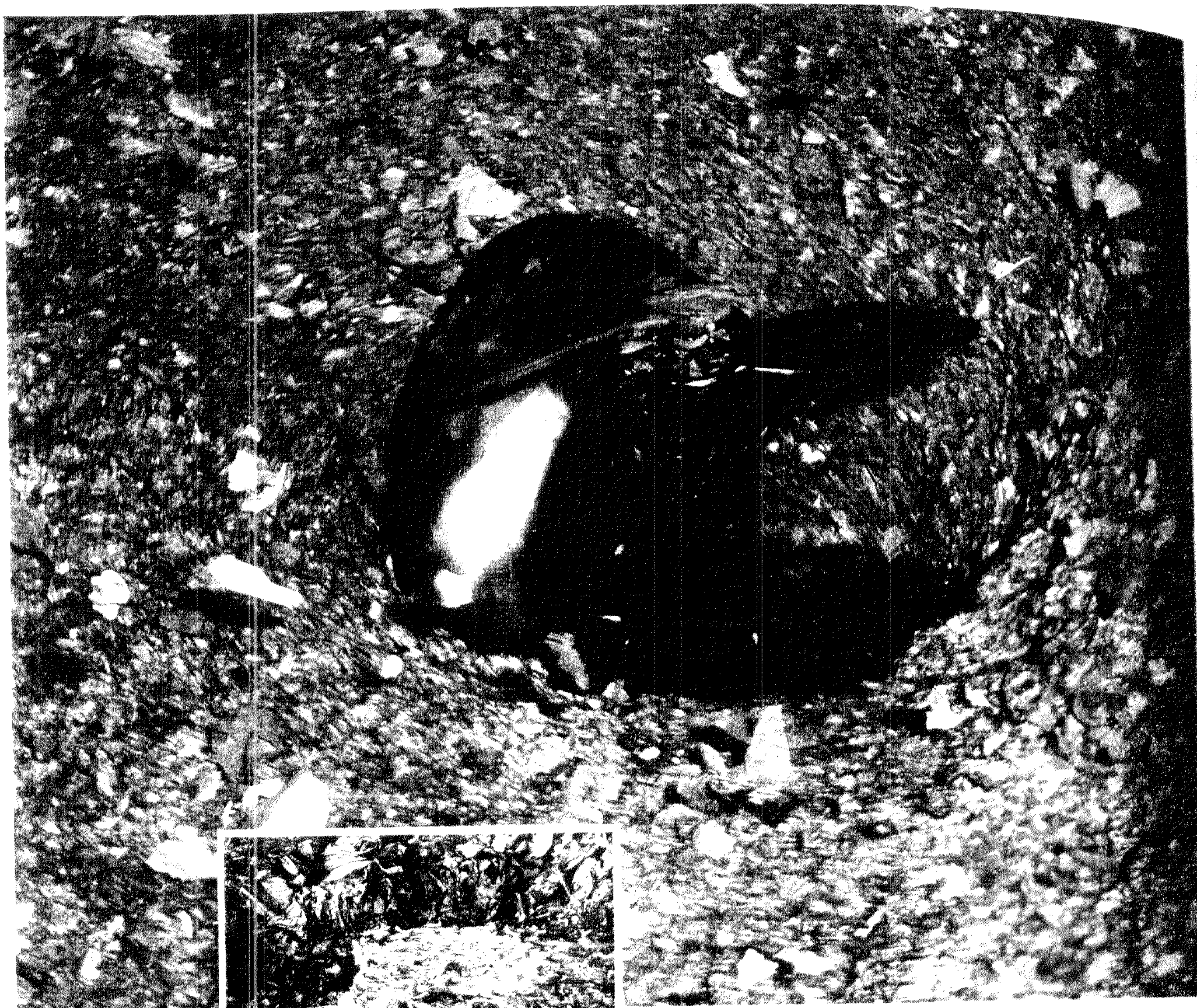
Chapters

1. Introduction

The extent to which deinked fibers are bleached depends on the raw material and the brightness requirement for the final product. Bleaching is conducted by either the oxidation or reduction of the chromophores in the fibers. Chemical pulps are bleached in delignifying multi-stage processes with strong oxidizing agents like oxygen, chlorine dioxide and hydrogen peroxide. Delignifying bleaching steps cannot be applied on mechanical pulps because of their high lignin content. Generally, the recycling of wastepaper for printing, writing and tissue grades, includes an oxidative bleaching step with hydrogen peroxide. The additional application of formamidine sulfinic acid (FAS), a strong reductive bleaching agent, can produce a further increase in brightness and thereby improve the quality of the final product.

Unloading of wastepaper

Wastepaper storage



…ping of
…paper

Mixed office waste-paper, a high quality fiber source

2. Bleaching with formamidine sulfinic acid (FAS)

Bleaching with formamidine sulfinic acid (FAS) yields the best results under alkaline conditions. FAS is only poorly soluble in water (~ 27 g/l). Under alkaline conditions up to 100 g/l are soluble as sulfinate. While the sodium salt of FAS is highly soluble, it is also very unstable. Consequently the solution cannot be stored for a prolonged period. Therefore, alkaline bleaching solutions of FAS should be prepared continuously on-site and feed directly to the bleaching process.

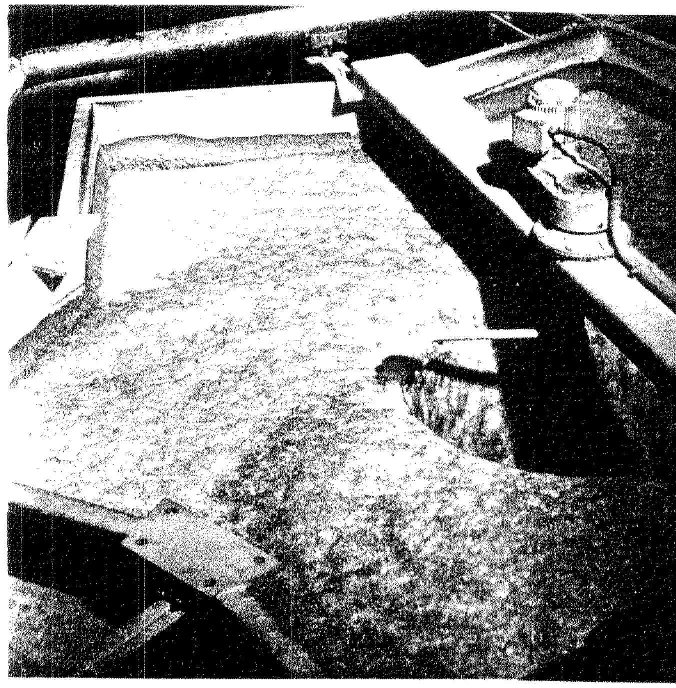
The equipment for the continuous preparation of the bleaching liquor is manufactured and supplied by Degussa.

Sodium sulfinate is oxidized during the bleaching process to urea and sodium bisulfite [1]. Sodium bisulfite is further oxidized to sodium bisulfate [2].

$$\begin{matrix} H_2N \\ HN \end{matrix}\!\!>\!C-S\!\begin{matrix} {}^{\nearrow}O \\ {}_{\searrow}OH \end{matrix} + NaOH \dashrightarrow \begin{matrix} H_2N \\ H_2N \end{matrix}\!\!>\!C=O + NaHSO_3$$

[1]

[2] $NaHSO_3 \dashrightarrow NaHSO_4$



...flotation cell for ...ink removal

The optimum weight ratio between formamidine sulfinic acid and caustic soda is close to 2:1. If the bleaching conditions are optimal, the initial pH of the wastepaper mixture is approximately 9 and decreases to a final pH between 7 and 8.

3. Application of FAS in Paper Recycling

The application of secondary fibers for the production of paper products has increased with the amount of deinked wastepaper growing at ever increasing rates. Only if the printing ink is almost completely removed the term "recycling"-process can be properly used. If the paper is reused without a deinking process, and the printing ink is only distributed throughout the paper sheet, the paper quality is reduced. This process can be discribed as a "downcycling"-process. "Downcycled" pulp is significantly lower in value and only of limited applicability due to its grey color shade.

Generally, printing ink is removed by either flotation or washing. Deinking by flotation exploits the differences in the hydrophobic/hydrophilic character of printing ink/fibers properties. In the flotation process the application of caustic, hydrogen peroxide, soda, sodium silicate and surfactants removes the printing ink from the fibers. The calcium soaps then collect the printing ink and are removed with the froth after aeration of the fiber suspension. Deinking by washing removes the printing inks in a process which is similar to the laundering of textiles. The fiber-printing-ink-suspension is dewatered on a sieve which simultaneously removes the printing inks, fillers and fines.

Residual printing inks mask the bleaching effect achieved in the fibers. **Only if the printing ink is almost completely removed, an additional bleaching step, either oxidative or reductive becomes efficient.** Improving the brightness by an oxidative postbleaching with hydrogen peroxide is described in detail in the Degussa brochure "Hydrogen Peroxide – Application in Deinking Processes". Paper dyes are generally not bleachable with hydrogen peroxide.

3.1 Pulper Bleaching with FAS

A very simple application of FAS is its addition with caustic soda directly into the pulper. This application is very efficient if wastepaper with high amounts of colored broke, massdyed wastepapers, or carbonless copy papers are recycled.

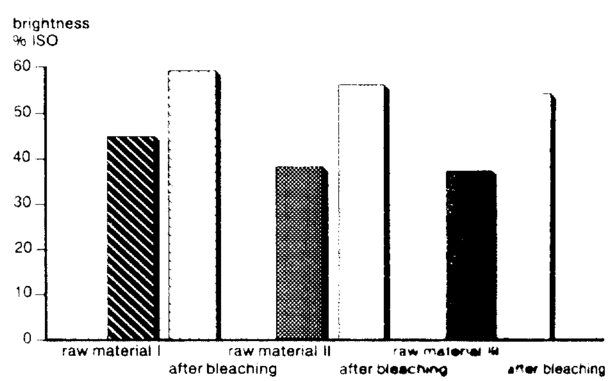


Figure 1
[illegible]ing of carbon-
[illegible] paper broke
[illegible]AS in the pulper;
[illegible] 0.8 % FAS,
[illegible] NaOH, consis-
[illegible] 55 °C,
[illegible] retention time.

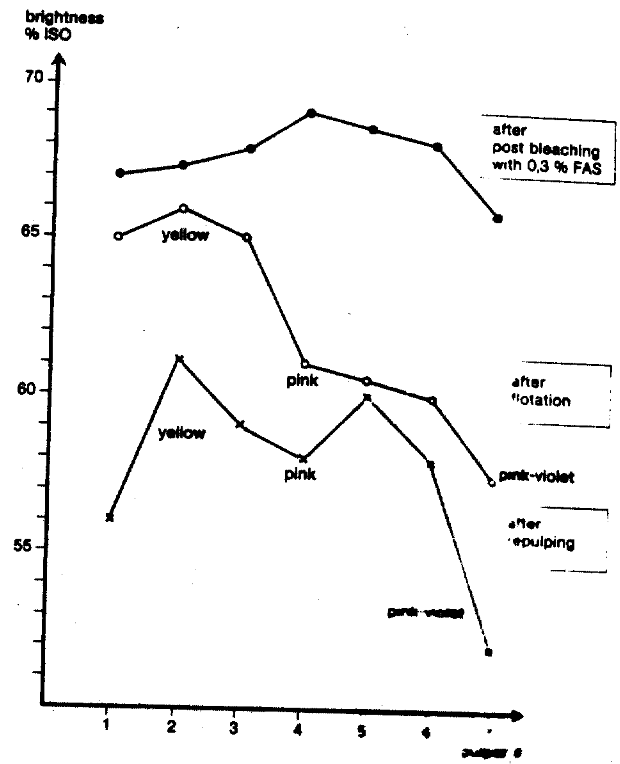
The maximum brightness gain is limited by the amount of printing ink in the wastepaper and the type of dyestuff used. The more printing ink the wastepaper contains, the more the bleaching effect of FAS is masked. **Figure 1** illustrates the bleaching effect of FAS on three different mixtures of broke from the production of carbonless copy paper.

Formamidine sulfinic acid is, like other reductive bleaching agents, attacked by oxygen. During repulping, atmospheric oxygen can react with FAS and decrease its efficiency.

3.2 Postbleaching with FAS

Postbleaching with FAS in a bleaching tower inhibits the inclusion of air at low and medium consistency, thereby improving the efficiency of FAS.

In practice, final pulp brightness and pulp color shade are important. **Figure 2** illustrates these effects using mass dyed deinked pulp which was treated with hydrogen peroxide in the pulper. The brightness was determined after the pulper, after flotation, and after the reductive post-treatment. Pulp brightness varied widely after repulping. The flotation stage after repulping only partially compensated for this effect. Reductive postbleaching with only 0.3 % FAS produced a more stable brightness and at the same time eliminated the color shades.



[illegible] 2
[illegible] bleaching of par- [illegible] mass dyed waste- [illegible] with 0.3 % FAS. [illegible] and deink- [illegible] wastepaper [illegible] quality with [illegible] 0.7 % H_2O_2 [illegible] standard deinking [illegible]

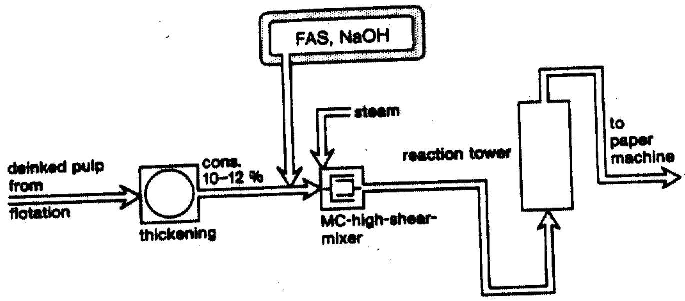


Figure 3
Application of formamidine sulfinic acid in postbleaching of wastepaper pulp at consistencies between 3–12% and an upflow bleaching tower.

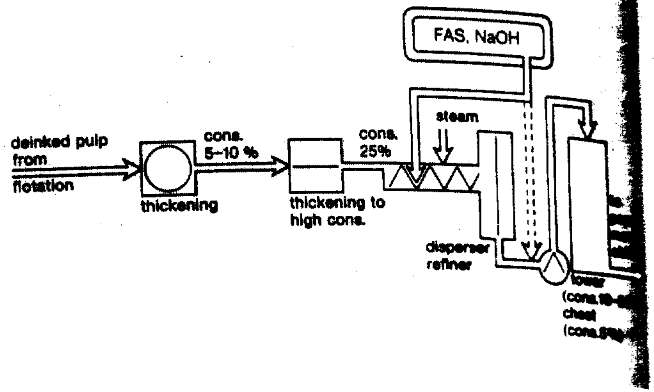


Figure 4
Flow diagram of hot disperser bleaching with FAS

Figure 3 illustrates a postbleaching application with FAS.

The bleaching conditions for formamidine sulfinic acid can vary within a wide range. Bleaching can be conducted at low, medium and even high consistency in either an upflow or downflow tower. Depending on the equipment, a tube or a chest can be used in the consistency range of 3–12%. However, to maximize the bleaching efficiency of FAS, high temperature or a long retention time is required. High consistency bleaching must be conducted at high temperature with high intensity mixing. These two conditions permit rapid bleaching under conditions which minimize oxygen interference.

In general, the following bleaching conditions can be employed.

Low consistency:	Consistency 3–5%, temperature 50–80 °C (120–175 °F), retention time 1–3 h.
Medium consistency:	Consistency 10–20%, temperature 50–80 °C (120–175 °F), retention time 1–2 h.
High consistency:	Consistency 25–35%, temperature 80–120 °C (175–250 °F), retention time 0.25–1 h.
High intensity mixing: (Hot disperser bleaching)	Retention time several minutes, post reaction at lower consistency, temperature 40–80 °C (110–175 °F).

An example of high consistency bleaching in a hot dispersing unit is illustrated in **Figure 4**. FAS is metered together with caustic soda into the heating screw of the dispersing unit at high consistency. Hot dispersing is followed by a subsequent reaction at low consistency with at least 15 min retention time.

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The addition of the FAS bleaching solution in front of the hot dispersing unit ideally combines the benefits of high intensity mixing with the high temperature. If the disperser is followed by a chest with a retention time of 15–30 min, a special bleaching tower for the FAS posttreatment is not required. The temperature in the dispersing unit should be above 90 °C (190 °F). Thus, the FAS posttreatment can be applied in an existing deinking plant with only a small capital expenditure.

3.3 Bleaching results

As a result of the heterogeneous nature of wastepaper, pulp produced from this raw material shows significant brightness variation after repulping, cleaning and bleaching. The objective of postbleaching is to brighten and eliminate the brightness variation. This effect was already illustrated in **Figure 2.**

Even though flotation or wash-deinking steps have removed the printing ink from the fibers, residual dyes cause coloration of the wastepaper pulp. These paper dyes are very stable against oxygen based bleaching agents like hydrogen peroxide. As a result, hypochlorite was widely used to destroy these dyes.

The two main reasons for the declining use of hypochlorite for dye removal are: 1) The delignifying ability of hypochlorite is not compatible with mechanical pulp fibers. It causes yellowing of the pulp fibers. As it becomes more and more difficult to process wastepaper which is free of mechanical fibers, highly bleached CTMP, and similar high yield pulps, the use of hypochlorite will diminish. 2) The application of hypochlorite produces halogenated compounds, e. g. chloroform. The environmental impact of these compounds will continue to limit the use of chlorine containing bleaching agents.

Reductively destroying paper dyes is thereby the method of choice to avoid the aforementioned problems. The high reducing power of FAS makes it an outstanding chemical to bleach a large number of today's dyestuffs.

The efficiency of postbleaching with FAS depends on the bleachability of the dyes (pigment dyes like phthalocyanin derivatives are not bleachable with reducing agents), the amount of massdyed paper, and the proportion of mechanical pulp. Wastepaper mixtures dedicated for the production of tissue paper normally contain only small amounts of mechanical pulp. The effect of color variation and brightness increase can be especially significant in these cases. Wastepaper with a high level of mechanical fibers for the production of printing and writing grades, for example, normally responds less to a post-bleaching stage. The brightness increase in these cases may be limited.

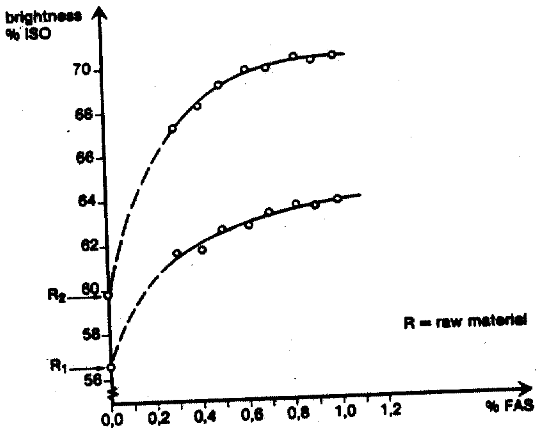

Figure 5
Post bleaching of two different wastepaper mixtures deinked by flotation with FAS at 70 °C (160 °F), 4 % consistency, 2 h retention time, FAS: NaOH ratio of 2 : 1.

Figure 5 illustrates the effect of FAS charge in the postbleaching of a deinked wastepaper mixture with a low level of mechanical fibers.

The effect of FAS tower bleaching on carbonless copy paper broke (wood-free massdyed paper) is illustrated in **Figure 6**.

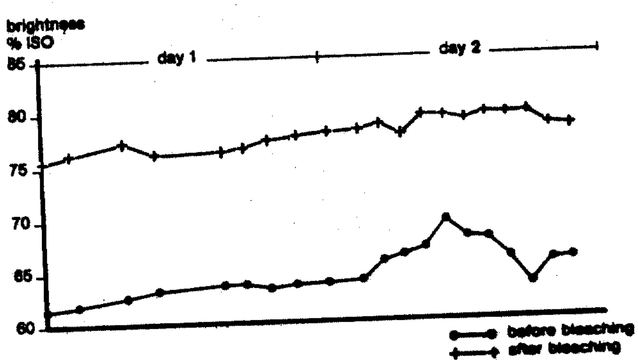

Figure 6
Mill data comparing the brightness before and after FAS bleaching. Final product: Tissue grades, conditions: 0.4 % FAS, 0.2 % NaOH, 12 % consistency, 60 °C (140 °F), 2 h retention time.

As described in Chapter 2 the ratio of caustic soda to FAS is important for efficient bleaching. An example of the results obtained with increasing amounts of caustic soda is illustrated in **Figure 7**.

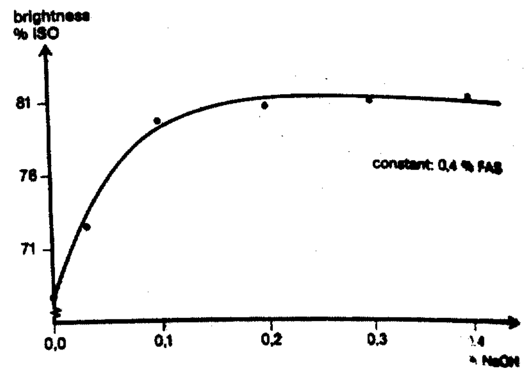

Figure
FAS postbleaching of wood free deinked pulp. Effect of the caustic soda charge at a constant charge of 0.4 % FAS. Bleaching conditions: 4 % consistency, 70 °C (160 °F), 2 h retention time.

If wood containing wastepaper is bleached, the caustic soda charge has to be carefully controlled and optimized for each application. If the alkalinity is too high, an irreversible alkaline yellowing of the fibers may result.

The speed of the brightness increase is dependent on the bleaching temperature. Very high bleaching temperature results in complete consumption of the formamidine sulfinic acid within several minutes and a concomitant brightness increase.

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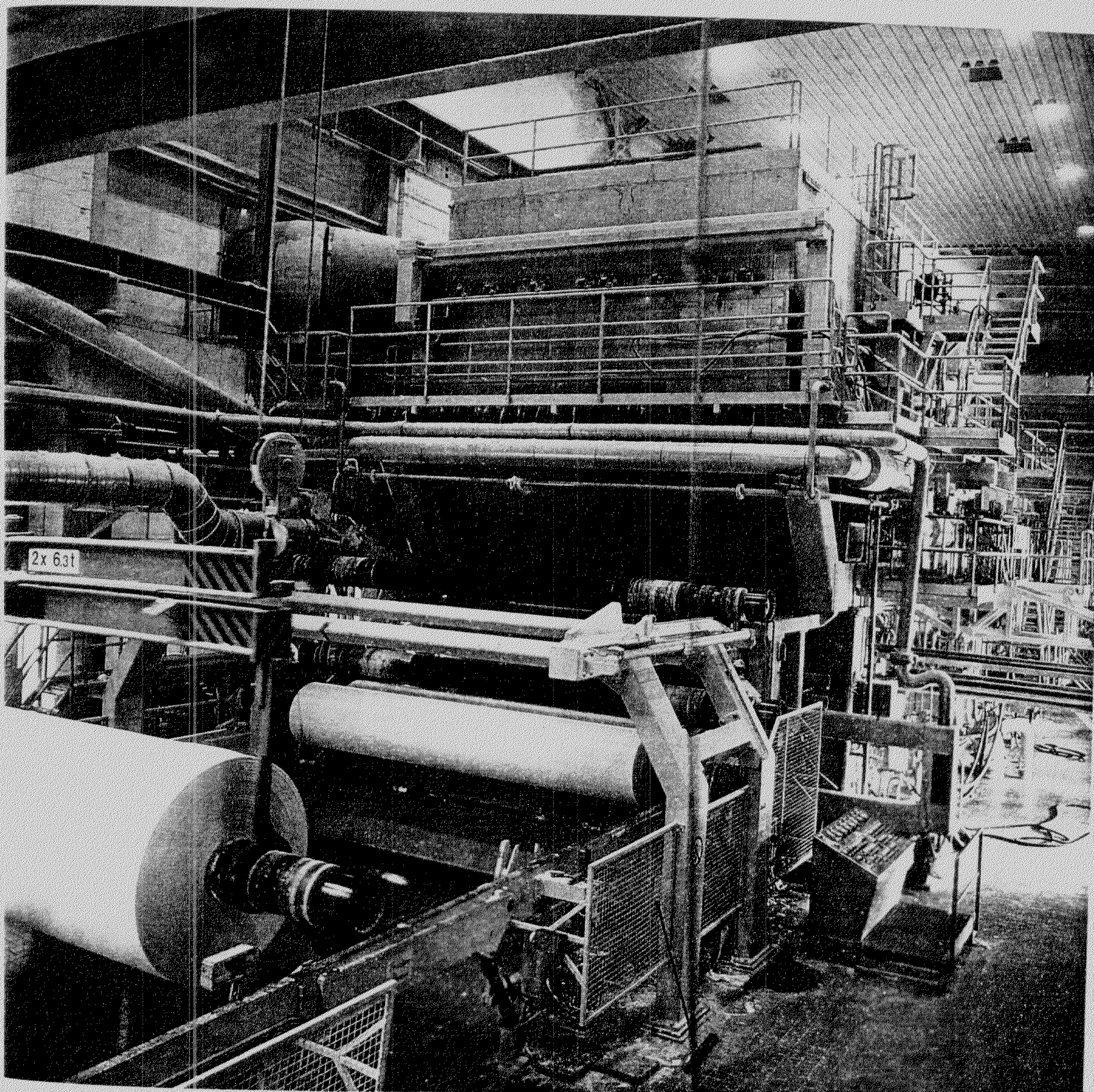


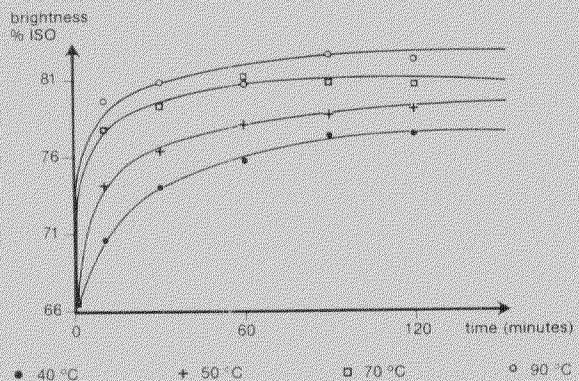
Paper machine, producing tissue paper from FAS bleached, recycled fibers

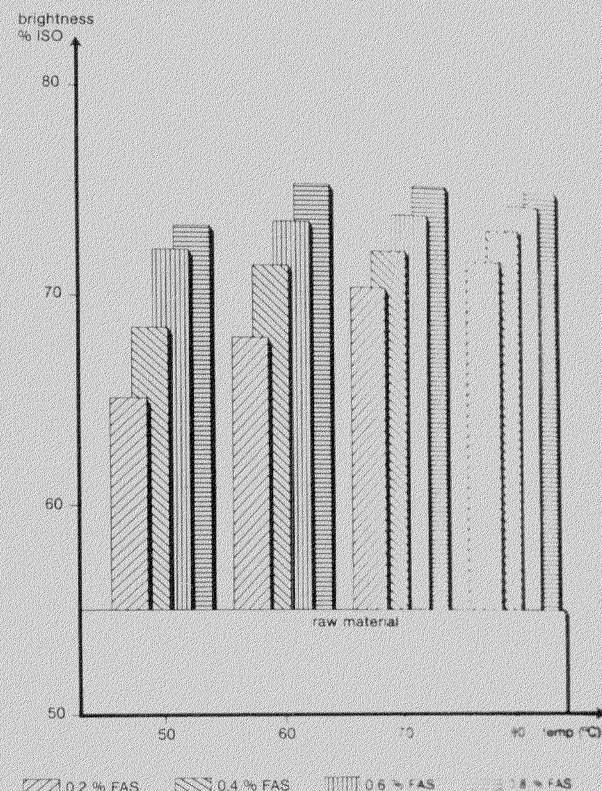
Figure 8
Postbleaching of wood free deinked pulp with 0.4 % FAS, 0.2 % NaOH and 4 % consistency. Effect of temperature and retention time on brightness increase.

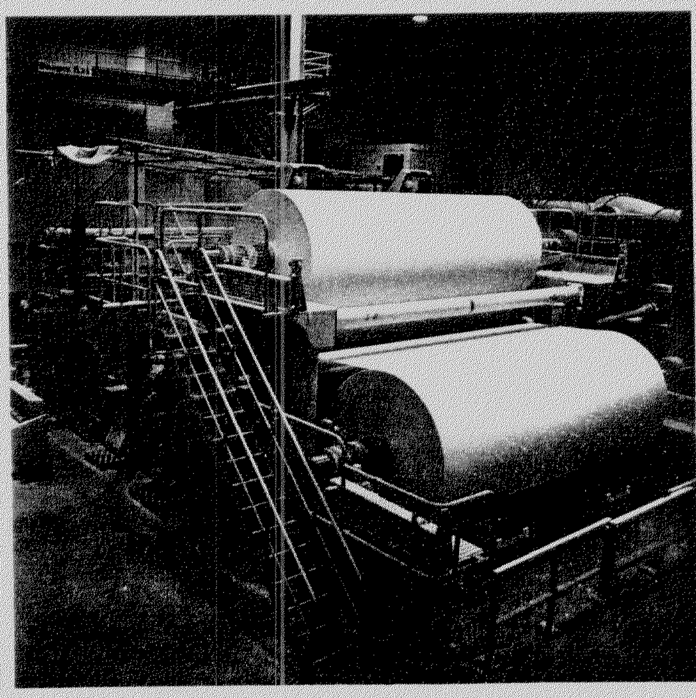
An example of the effect of reaction temperature and retention time is illustrated in **Figure 8**.

As expected, with an increasing amount of FAS the brightness increase becomes more pronounced. The positive effect of increased temperature bleaching chemical demand is illustrated in **Figure 9**. High bleaching temperature saves bleaching chemical.

Figure 9
Effect of bleaching temperature in postbleaching of wastepaper pulp with different amounts of FAS. The ratio of FAS to caustic soda held constant at 2:1.







Winder

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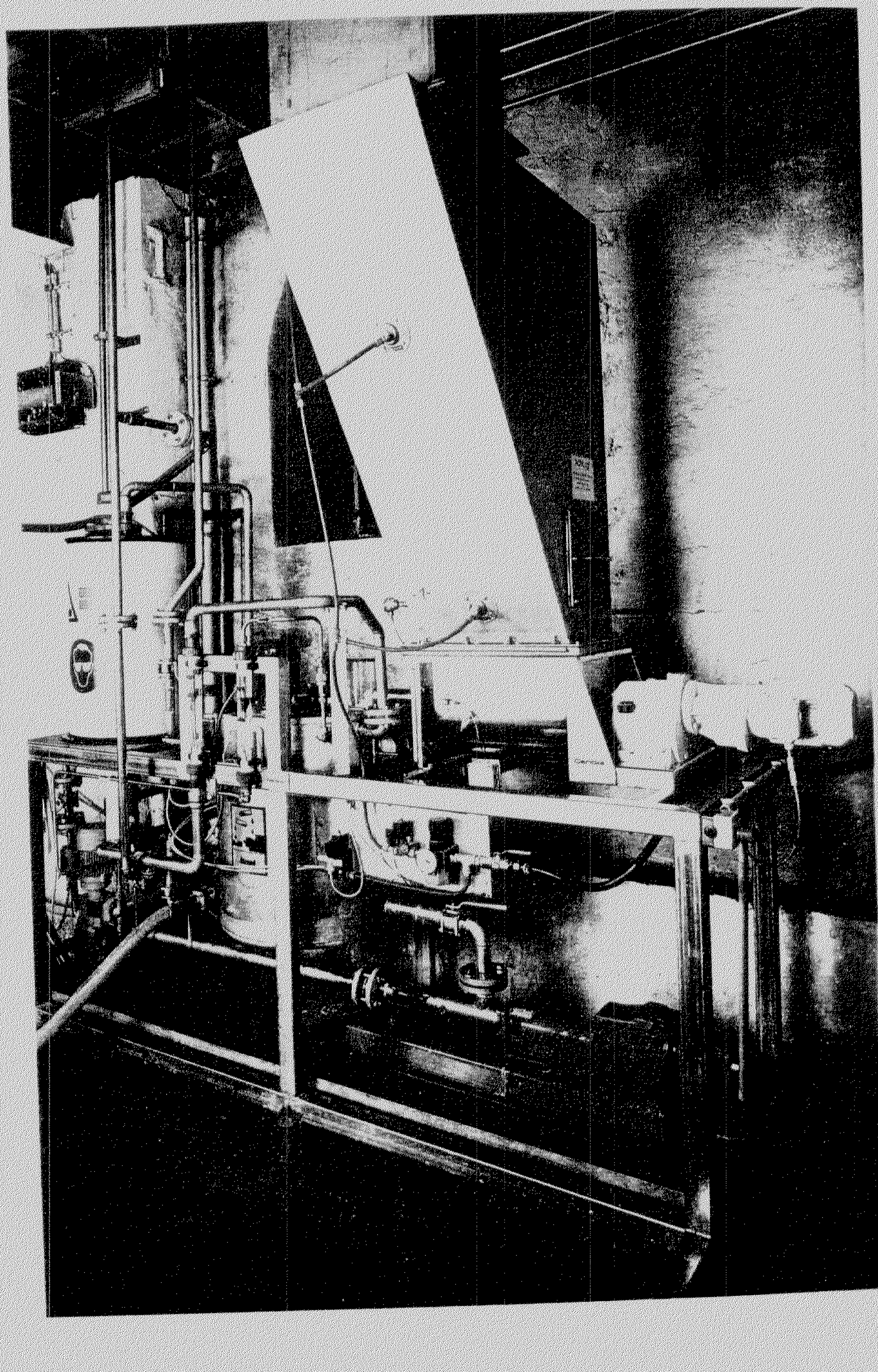
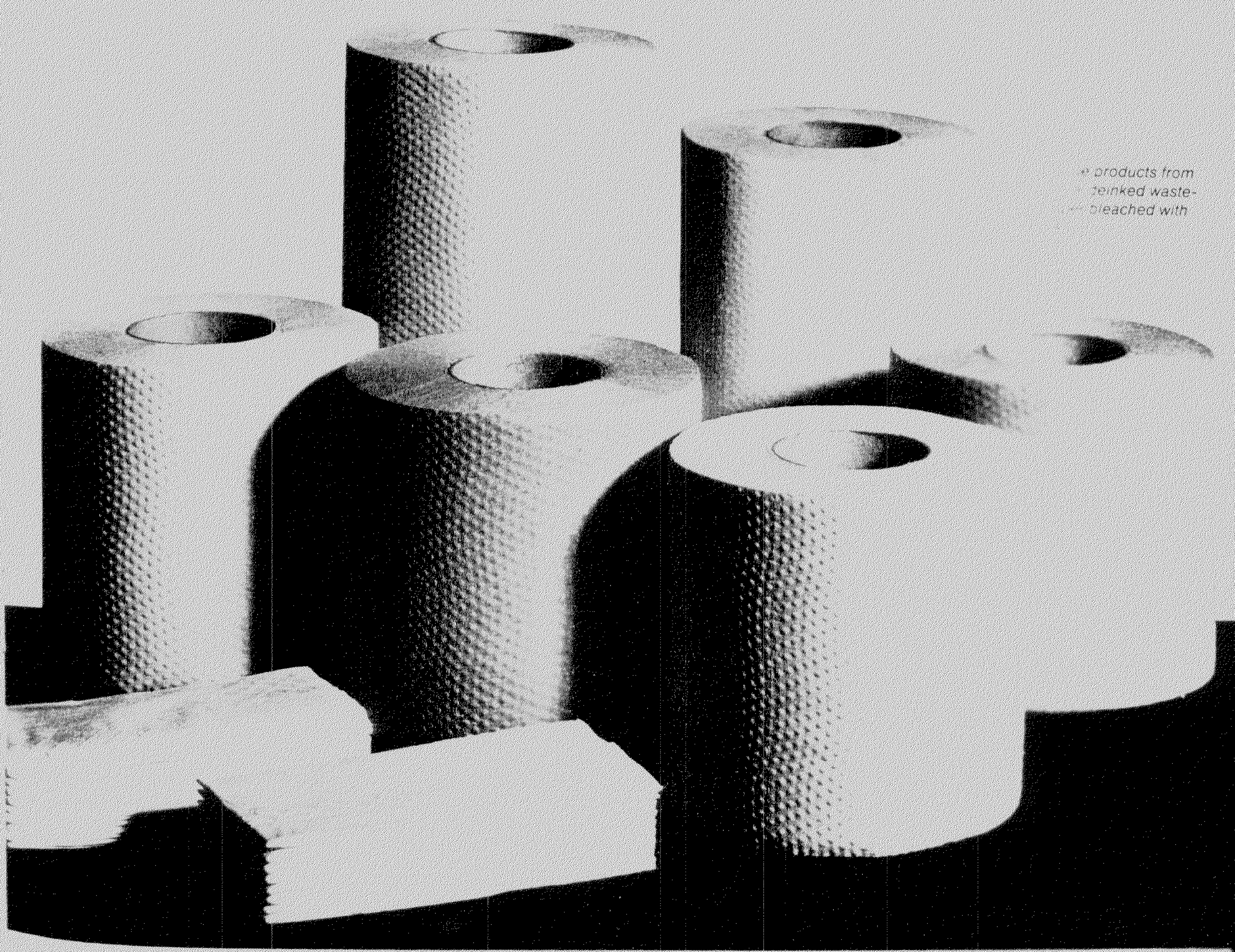
4. Product information

FAS	Formamidine sulfinic acid Odourless, stable reductive bleaching agent
Areas of application:	Postbleaching of deinked materials
Chemical formula:	$H_2N - \underset{\underset{NH}{\Vert}}{C} - SO_2H$
Molecular mass:	108.12 g/mole
Appearance:	White to light yellow, crystalline powder
Purity:	Minimum 98 % formamidine sulfinic acid
Solubility:	27 g in 1 litre of water at 20 °C (alkaline solutions, up to 100 g/litre can be dissolved at room temperature. These solutions have a low shelf live.)
Decomposition point:	123 °C (differential thermal analysis at a heating rate of 3 – 4 °C/min)
pH:	approx. 4.0 (1 % aqueous solution)
Test methods:	a) Titration of alkaline solutions with potassium bichromate against dimethyl-4'4-bipryridinium dichloride as an indicator. b) With vat paper, alkaline solutions at 70 °C or higher result in a blue coloration in the presence of active FAS; this takes on a greenish tinge if quantities are low (under 0.3 g/litre)
Handling and storage:	Store in a cool, dry place at ambient temperature, recommended storage temperature 25 °C – 35 °C (75 °F – 95 °F) Keep away from concentrated alkalis and and oxidizing agents
Bulk weight:	Approx. 0.85 kg/litre
Packing:	Fibre drums with polyethylene inner sacks; 50 kg net each, super sacks 500 kg
Brussels customs tariff:	2931
Risks:	The complete information for product safety and handling is available in the DIN safety data sheet (MSDS). This product is harmful if swallowed, and irritates skin and eyes. If exposed to heat the product will decompose spontaneously in an exothermic reaction, producing sulphur dioxide (SO_2) and ammonia sulphate ($(NH_4)_2SO_4$)



Dosing unit for the continuous production of alkaline FAS solutions. The FAS is fed from 50 kg drums or 500 kg super sacks from the floor above.

Protective measures:	When handling the solid product and its solutions, suitable protective gloves and goggles/mask should be worn. If the product decomposes, flood with large amounts of water; wear a respirator
First aid:	If skin comes into direct contact with the product, rinse off thoroughly with water. If swallowed, contact a physician immediately (symptomatic therapy)



...e products from ... deinked waste- ... bleached with

Tissue products made from deinked waste-paper bleached with FAS

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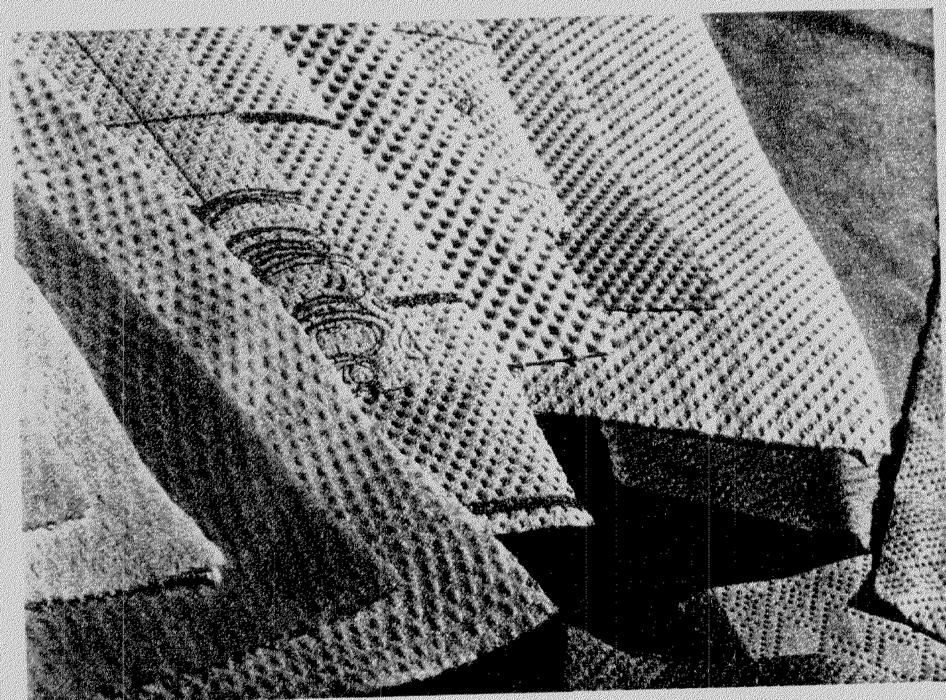

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ADVANCED BLEACHING TECHNOLOGY FOR SECONDARY FIBERS

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ABSTRACT

In addition to the recycling of ONP and magazines, other printing and writing papers, especially office waste and household collections, are increasingly being included in the recycling loop.

Conventional bleaching systems for deinked pulps are limited in their ability to cope with "difficult-to-recycle wastepaper" and environmental regulations at the same time. New bleaching equipment, chemicals and strategies are required.

Fortunately, a great deal of progress has been made in deinking and bleaching technology. New concepts, such as disperser bleaching and separate bleaching of long and short fiber fractions, are described by means of lab and mill data.

The benefits of these new approaches are lower chemical demand, higher achievable brightness levels, efficient color stripping and more.

ADVANCED BLEACHING TECHNOLOGY FOR SECONDARY FIBERS

I. INTRODUCTION

The increased usage of wastepaper for all end-use applications has provided the impetus for developing new technologies to upgrade its quality. Existing technologies to help achieve this goal include pulper bleaching with hydrogen peroxide (H_2O_2), single-stage post-bleaching with either hydrogen peroxide, sodium hydrosulfite (Y), or both in a two-stage process.

To meet the challenges of producing higher brightness, whiter pulp, the use of reducing agents such as formamidine sulfinic acid (FAS) is playing an increasingly important role to help meet these objectives. The greater redox potential and thermal stability of FAS make this chemical ideal for bleaching applications ranging from conventional post-bleaching to hot (thermal) disperser bleaching.

This paper will review the different pulp bleaching strategies with respect to bleaching chemicals and their applications.

II. Bleaching Chemicals

Chlorine containing bleaching chemicals such as Cl_2, ClO_2 and NaOCl are no longer acceptable for the bleaching of secondary fibers because of their economical and ecological impact (1). The only acceptable oxidative bleaching agents are oxygen and hydrogen peroxide. In addition, the reductive bleaching chemicals sodium hydrosulfite and FAS are being used in the industry. Table I lists these chemicals with their redox reaction and redox equivalents.

A) Oxidants (O_2 and H_2O_2)

Oxygen is a widely used oxidant to delignify and bleach chemical pulps. In contrast, bleaching of recycled wastepaper with oxygen is not used at all in the industry. Results of a pilot scale oxygen bleaching system of recycled pulp have shown that oxygen can be useful to improve brightness, cleanliness and to remove stickies of OCC (2). In the case of bleaching mechanical pulp containing wastepaper, oxygen bleaching does not seem to be economical because of high capital cost, low bleached pulp yield, and its poor bleaching efficiency.

Today, the most widely used oxidative bleaching agent in deinking mills is hydrogen peroxide. Hydrogen peroxide is easy to handle, is applicable over a wide range of reaction conditions and is one of the most environmentally compatible bleaching agents. The economy of its application though, depends greatly on the specific optimization of addition points in the deinking plant, chemical charges, and reaction conditions.

B) Reductants ($Na_2S_2O_4$ and FAS)

Zinc/sodium hydrosulfite (Y) has been used for bleaching for about 30 years (3). Depending on the reaction conditions, hydrosulfite is more or less oxidized to sulfate, sulfite and thiosulfate during the reaction. Oxygen and air easily oxidize hydrosulfite (3) and can reduce its efficiency.

FAS (formamidine sulfinic acid) has been successfully used in some deinking mills during the past years. During bleaching, FAS is mainly oxidized to sodium sulfate according to the reaction equation shown in Figure 1. FAS is generally activated with caustic soda to a degree that the final pH of the bleaching reaction is in the range of pH 7-8.

III Deinking and Bleaching Processes

Deinking of wastepaper consists basically of three process steps which differ from each other in their principle objective. These are firstly, the defiberization i.e. repulping and fiber swelling, secondly, the removal of larger impurities (mostly of non-fibrous nature) by mainly mechanical forces and thirdly, the ink removal through chemi-mechanical treatment by washing and/or flotation.

A) Current Deinking Concepts

A schematic of current deinking technology is depicted in Figure 2. Wastepaper, together with chemicals is repulped either batchwise in a hydropulper or in a continuously operated drum pulper. Both are followed by a reaction chest at approximately 5% consistency which allows the fibers to swell. Subsequently, the pulp is further diluted to low consistency, screened and cleaned before the pulp enters the actual deinking step. Deinking is achieved by either flotation, washing or by a combination of both. An excellent overview of all deinking steps has been given e.g. by D. R. Crow and R. F. Secor (4).

Cleanliness and brightness of pulp being deinked in such a "standard" deinking process can be further improved by adding a post deinking stage. A complete conventional deinking process including post-deinking and bleaching is schematically illustrated in Figure 3. After thickening the primary deinked pulp it enters a disperser in which a majority of the residual ink particles are broken down to sizes below the threshold of visibility and dispersed throughout the pulp. The remaining ink particles which are still visible can be more easily removed after such a treatment in a post deinking step by either flotation or washing.

B) Fiber Fractionation of Deinked Pulp

L. Floccia (5) has shown with results from mill experience that further improvements of deinked pulp could be achieved by fractionation of the deinked fibers with subsequent bleaching. W. Eul et al. (6) described the advantages of fiber fractionation of deinked pulp relative to the efficiency of bleaching the long and short fiber fractions separately. The advantages of fiber fractionation are mainly a higher achievable brightness for one fiber fraction and more economical use of bleaching chemicals. Figure 4 illustrates this process scheme for the fiber fractionation of deinked pulp. This approach is viable only if there is a use for the lower brightness fines fraction.

C) Fiber Fractionation prior to Flotation

While fiber fractionation of deinked pulp can improve the efficiency of bleaching chemicals it does not further improve the deinking capacity per se. A new deinking concept has been suggested and described in detail by W. Eul (1). The concept suggests fiber fractionation prior to flotation as shown in Figure 5, and can require as little as 50% of the flotation capacity of conventional deinking systems to produce pulp of the same final brightness. New concepts like this which emphasize efficient deinking and "selective" bleaching are a prerequisite for the further improvement of bleaching efficiency and to maintain the value of the recycled fibers.

IV Application of Bleaching Chemicals

A) Standard Bleaching Practices

1. Pulper Bleaching with H_2O_2

The use of H_2O_2 in the pulper can improve the final pulp brightness and inhibit the yellowing reaction which can occur with mechanical pulp fibers under alkaline conditions. The pulping conditions and chemical charges which can fully exploit the properties of H_2O_2 for pulper bleaching are: 15-20% consistency, 40°C for 20 minutes repulping time, and 60 minutes reaction time at 4 to 5% consistency in a reaction chest at 40°C. The typical chemical charges are: 1.0% H_2O_2 (100%), 1.0% NaOH (100%), 2.0% sodium silicate (38-41° Bé), 0.2% Na_5DTPA (40%), 0.5-1.0% fatty acid, soap, or other collector. Additionally, individual mill conditions may dictate other dispersers/emulsifiers or talc for stickies removal. These chemical charges are well established and have been used in many mills, and also laboratory conditions. The effect of H_2O_2 in the pulper on brightness of various wastepaper grades is illustrated in Figure 6.

The most important variable for achieving an improved brightness response from H_2O_2 pulper bleaching is that of high consistency. The effect of stock consistency on brightness after flotation is illustrated in Figure 7. High consistency conditions result in a higher ratio of bleaching agent to fibers, while lowering the concentration of H_2O_2 decomposing substances. Additionally, the higher friction due to fiber-to-fiber interactions at high consistency improve the printing ink separation.

2. Tower Bleaching

Oxidative post-bleaching with H_2O_2 is performed as illustrated in Figure 8. Bleaching with H_2O_2 increases the brightness without necessarily eliminating the color shade which may be present. Typical chemical charges are: 1.0% H_2O_2, 0.8% NaOH, 2.0% sodium silicate, 0.1% Na_5DTPA, 60°C, 20% consistency, 1-3 hours. Depending on the furnish composition concentration of stabilizers such as sodium silicate and/or DTPA could be reduced; as might be the case with a wood-free furnish, or increased in cases of furnish containing high levels of wood-containing wastepaper.

Reductive post-bleaching with sodium hydrosulfite (Y) or formamidine sulfinic acid (FAS) are illustrated in Figure 9. Reductive bleaching agents are capable to improve brightness and also removing color shades. Typical conditions for Y bleaching are: 1.0% sodium hydrosulfite, starting pH 6.0, 5.0% cons., 60°C, 1 hour; FAS bleaching are: 0.3-0.6% FAS, 0.15-0.3% NaOH, 5.0% cons., final pH 7-8, 70°C, 1 hour.

The comparative bleaching effects of H_2O_2, Y, and FAS are illustrated in Figure 10. On average, post-bleaching increases the brightness approximately four points.

Dyes which are resistant to decolorization with sodium hydrosulfite can very often be decolorized with formamidine sulfinic acid (FAS). Figure 11 shows the results of a plant trial with an FAS post-bleaching stage. Some deinked pulps were extremely colored even after flotation. As shown by the top line of Figure 11, no color shade was observed after the alkaline bleaching step with 0.3% FAS.

Figures 12, 13, 14 illustrate another study which compares post bleaching with Y and FAS. While both FAS and Y demonstrated color removal ability with subsequent brightness improved, FAS was superior in all cases.

As a consequence of environmental pressures which are restricting the use of chlorine containing bleaching chemicals, FAS can serve as a partial replacement for sodium hypochlorite (H). In a laboratory study deinked pulp was treated with up to 1.0% FAS and 4.0% NaOCl. The results are illustrated in Figure 15. The maximum brightness of 78 ISO for FAS was less than the 82 ISO brightness achieved with sodium hypochlorite. However, generally speaking, FAS still provides the best opportunity to produce non-chlorine bleached pulps in one stage of high brightness without potentially interfering color shades.

3. Two-Stage Bleaching

Combinations of peroxide and hydrosulfite bleaching stages have been used for both mechanical pulp bleaching (7) and post-bleaching of deinked pulp. Typical results obtained on deinked pulp with P-Y post-bleaching usually accounts for a 2-6 points brightness increase as a result of the second Y stage. Unpublished laboratory data has demonstrated replacing FAS for Y has yielded superior results with respect to brightness and color removal compared to the conventional P-Y sequence.

In another internal study, results with the "reversed" FAS-P sequence improved brightness an additional 8.0 points compared to the P-FAS sequence (Figure 16). This sequence may provide an opportunity to further increase quality of deinked pulp with respect to both brightness and color.

4. Bleaching Chemical Charge Optimization Between Stages

Internal research reports have evaluated the effects of distributing the H_2O_2 charge between the pulper and post-bleaching stages on brightness. Figures 17 and 18 illustrate the optimum brightness is obtained when 0.5% H_2O_2 is added to the pulper with up to a total of 3.0%

H_2O_2 applied. This usually implies that a minimum charge of H_2O_2 is required to prevent alkali yellowing and facilitates further bleaching in the pulper. This result may provide further opportunities to upgrade/economize the bleaching of recycled fibers.

B) <u>New Bleaching Practices</u>

1. Hot Disperser

The quality and appearance of deinked pulp is not only determined by its brightness and color but also its cleanliness. It is therefore essential for high-quality deinked pulp to be free of dirt specks and ink particles. One way to achieve a cleaner pulp is to destroy these impurities by means of a thermomechanical treatment, or as is more commonly called, a hot dispersion step. The dispersion step reduces the visible impurities into more numerous, smaller particles; and, without subsequent bleaching, yields a pulp with a grey-colored appearance.

Figure 19 illustrates the application of H_2O_2, Y, and FAS for disperser bleaching.

a. Bleaching with H_2O_2

The objective of bleaching with H_2O_2 in the disperser is to increase or maintain the brightness of the pulp after the thermomechanical treatment utilizing the disperser unit as a chemical mixer.

Bleaching with H_2O_2 in the disperser is accomplished by adding a solution of alkaline hydrogen peroxide prior to the disperser disc. Typical chemical charges are: 0.5-1.0% H_2O_2, 0.25-0.5% NaOH. In order to promote the highest bleaching efficiency, dilution water should be kept to a minimum to maintain the highest possible consistency in the dispersing zone.

Depending on deinked stock composition, brightness gains of 2 points can be achieved. If, however, lower than expected brightness is being achieved, it might be advantageous to add a stabilizer. Typically, sodium silicate is added to the bleach liquor at a charge of between 0.5-1.0%. The addition of silicates, however, can shorten the lifetime of the disperser discs by forming abrasive glass-like precipitates. Therefore, the cost benefit between silicate addition, higher H_2O_2

charges, and disc life need to be considered. Table II illustrates typical results obtained with H_2O_2 in a hot disperser.

b. FAS, Y

Bleaching in the hot disperser with FAS has several advantages over hydrosulfite and hydrogen peroxide. Firstly, the high temperatures and short reaction times are not comparable with sodium hydrosulfite, as decomposition can occur; FAS is stable at disperser temperatures of 90-130°C.

The bleaching reaction with FAS is 60-80% complete as the pulp exits the disperser. Consequently no additional pumps, mixer and tower are required. An additional one-half hour may be required in a reaction chest to fully complete the reaction. In addition to the ability of FAS to brighten, the higher reduction potential of FAS is able to decolorize heavily dyed pulp stock.

The choice of which bleaching agent to be applied in the hot disperser is determined by the wastepaper composition, reaction conditions available and the end product requirements.

The use of FAS should be most beneficial in cases where non-shaded higher brightness pulp is to be produced from highly colored pulp.

The use of H_2O_2 is most recommended in cases where the brightness of the pulp needs to be maintained after the dirt and residual ink particles are ground in the disperser and impart a grey shade to the pulp.

2. Bleaching of Fractionated Pulps

Fiber fractionation and separate post-bleaching of the long and short (fines) fraction is the most efficient way to utilize bleaching chemicals and for producing high quality pulp.

a. Long Fiber Fraction

The long fiber fraction exhibits excellent bleachibility with H_2O_2. Typically, 1% H_2O_2 applied can increase the brightness 4-7 points. And, as a result of the fractionation ratio, H_2O_2 usage can be reduced by 30-50%. Figure 20 illustrates the effects of fiber fractionation on brightness and chemical savings.

b. Short Fiber Fraction

The short fiber fraction contains a large proportion of stickies, dirt particles, and colored printing ink particles. As a result H_2O_2 shows only a very poor bleaching results. Reductive bleaching with FAS or sodium hydrosulfite results in a 3 to 4 point brightness increase. However, the high content of printing ink and dirt mask the bleaching effect.

Figure illustrates the effect of FAS/Y Bleaching on the Short Fiber (Fines) Fraction.

IV Summary

New deinking processes and technologies are providing opportunities to produce higher qualities of recycled products. These technologies enable the deinked pulp to be bleached to the highest obtainable brightness at the most economical cost.

The bleaching concepts can be summarized as follows:

- A one-stage deinking process can be improved by post-bleaching. The choice of either H_2O_2, FAS, or Y, needs to be individually determined, and is dependent on the raw material composition.

- A two-stage P-Y, P-FAS post-bleaching sequence can incrementally improve the brightness and color shade of deinked pulp.

- The FAS-P sequence has the potential of further improving the brightning response compared to the P-FAS or P-Y sequence.

- The addition of bleaching chemicals to a hot-disperser provides the opportunity to retain and/or improve the brightness of the pulp after treatment. Additionally, FAS has the ability to remove the color shade from dyed pulps.

- Bleaching either the short or long fiber fractions separately results in better bleaching response and chemical cost savings compared to the bleaching of unfractionated pulps.

Either one or a combination of these new or existing technologies should make it possible to produce high brightness deinked pulp to meet the present and future quality requirements.

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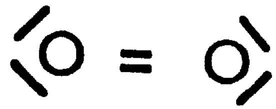
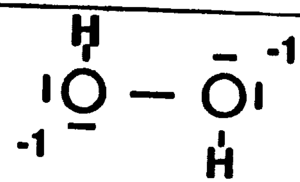
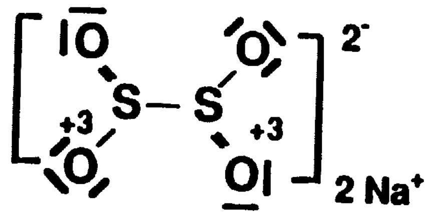
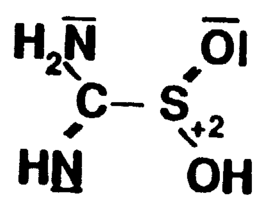
	Chemical Name	Structural Formula	Molecular Weight [g/mol]	Redox Reaction	Redox Equivalent e^-/mol
OXIDANTS	Oxygen		32.0	$O_2 + 2H_2O + 4e^- \longrightarrow 4OH^-$	4
	Hydrogen Peroxide		34.0	$HO_2^- + H_2O + 2e^- \longrightarrow 3\ OH^-$	2
REDUCTANTS	Sodium Hydrosulfite		174.1	$S_2O_4^{2-} + 4\ OH^- \longrightarrow 2\ SO_3^{2-} + 2\ H_2O + 2e^-$	2
	FAS (Formamidine Sulfinic Acid)		108.1	$(NH_2)\ NH\ C\ SO_2\ H^- + 6\ HO^- + \rightarrow (NH_2)_2CO + SO_4^{2-} + 3H_2O + 4e^-$	4

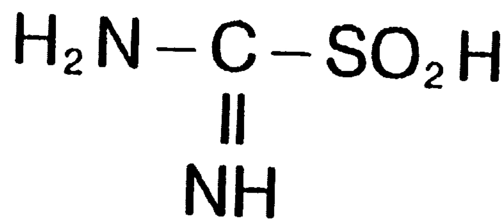
Table I

ENVIRONMENTALLY FAVORABLE CHLORINE-FREE BLEACHING AGENTS FOR WASTEPAPER RECYCLING PROCESSES

Bleaching Solution	Degree of Brightness % ISO		Brightness difference
	Before disperser	After disperser	
0.7% H_2O_2 0.1% NaOH 0.2% Stabilizer	52.5	53.6	+1.1
0.8% H_2O_2 0.3% NaOH Without Stabilizer	51.6	53.3	+1.7
0.7% H_2O_2 0.3% NaOH 0.2% Stabilizer	54.1	55.0	+0.9
0.5% FAS 0.1% NaOH	52.6	57.7	+5.1
0.5% FAS 0.1% NaOH	50.7	55.5	+4.8

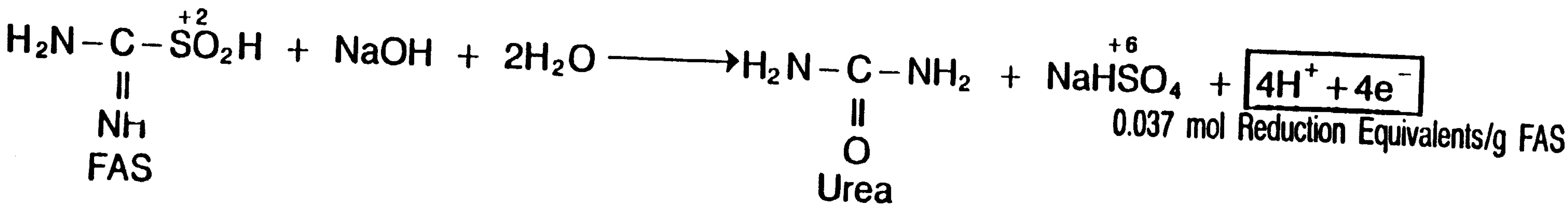
Table II

EFFECT OF THERMAL DISPERSER BLEACHING WITH FAS OR H_2O_2 ON BRIGHTNESS



108g/mol

<u>FAS – Bleaching Reaction</u>



ORP: 1% FAS – Solution, pH 10.0 : max –800mV

<u>Dithionite/Hydrosulfite – Bleaching Reaction</u>

$$Na_2\overset{+3}{S_2}O_4 + 4H_2O \longrightarrow 2\ Na\overset{+6}{H S}O_4 + \boxed{6H^+ + 6e^-}$$

0.034 mol Reduction Equivalents/g Dithionite

ORP: 1% Dithionite – Solution, pH6.0 : max –500mV

Figure 1

FAS – Formamidine Sulfinic Acid

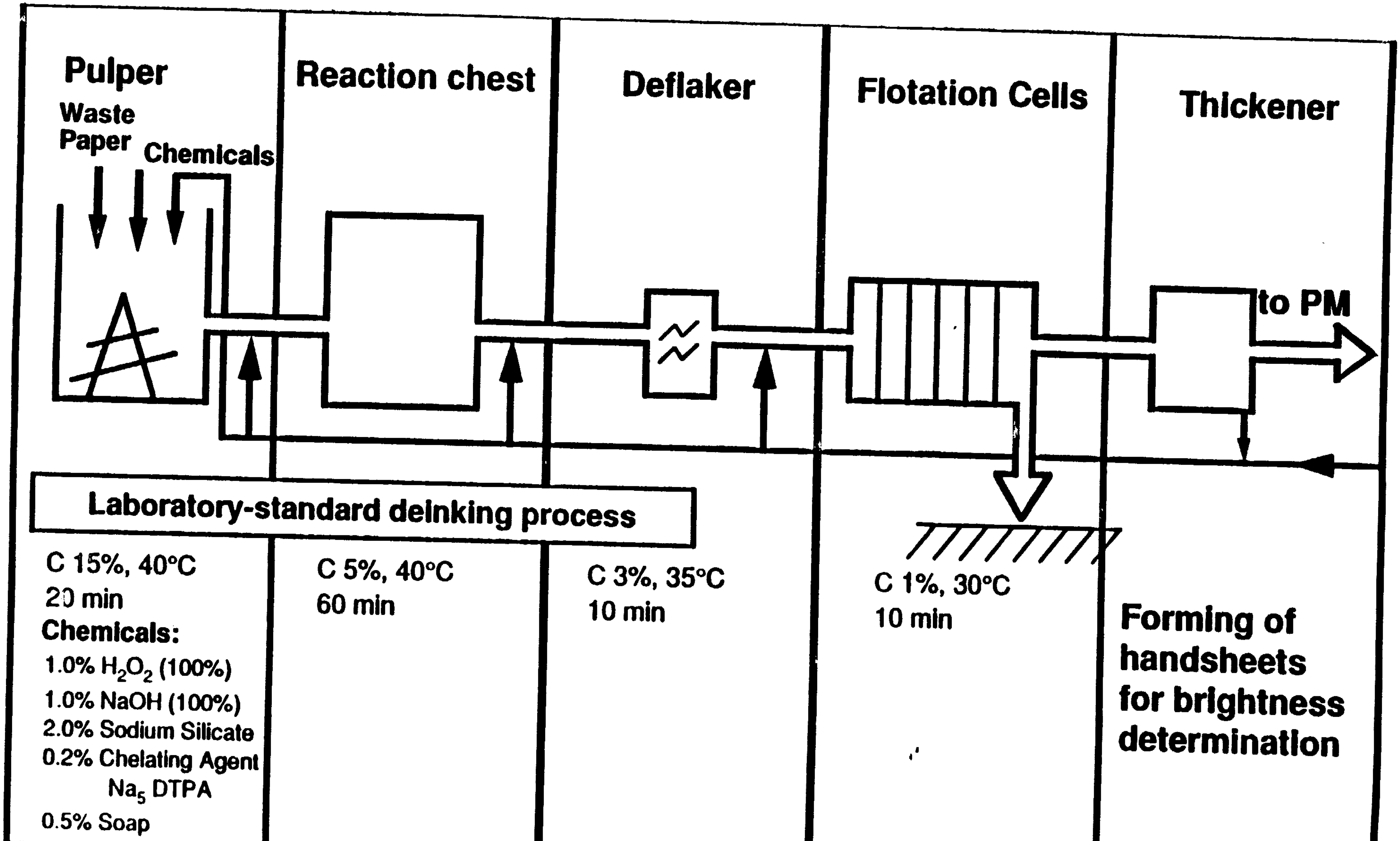
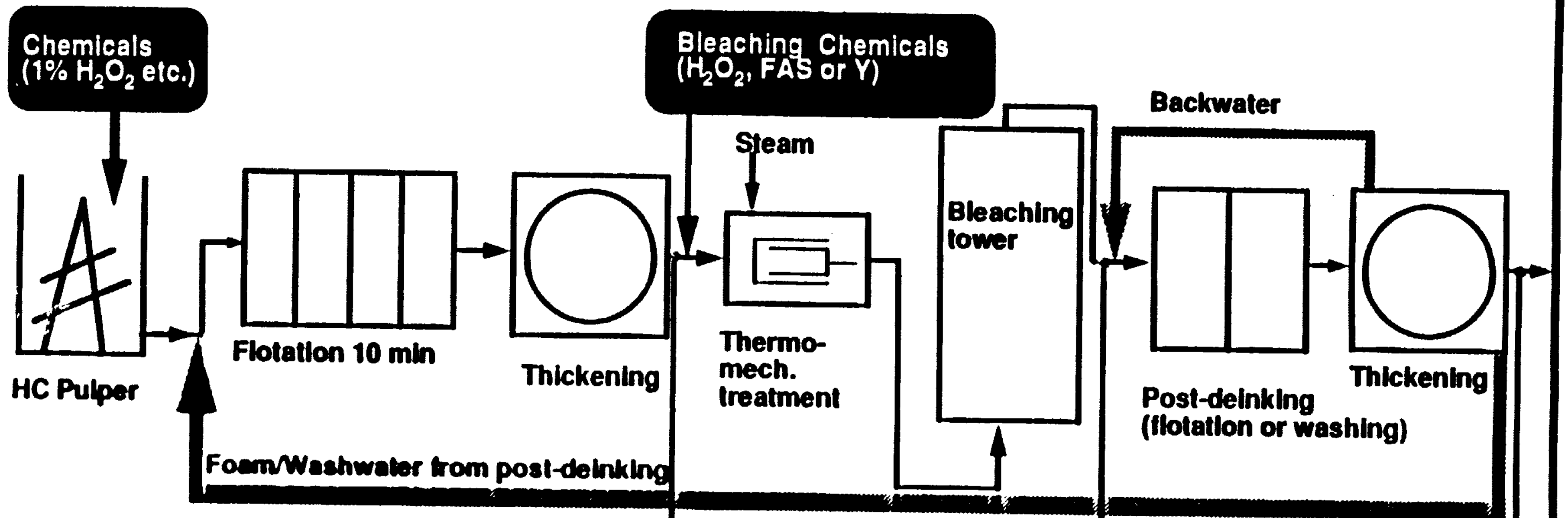


Figure 2

STANDARD DEINKING PROCESS WITH HIGH-CONSISTENCY REPULPING



	Raw material			
Np. (100%)	0 (47)	11 (58)	14 (61)	16 (63)
Np./Mag. (50/50)	0 (46)	15 (61)	18 (64)	20 (66)
Mag. (100%)	0 (43)	18 (61)	25 (68)	27 (71)
Wood-free	0 (57)	23 (80)	25 (82)	27 (84)

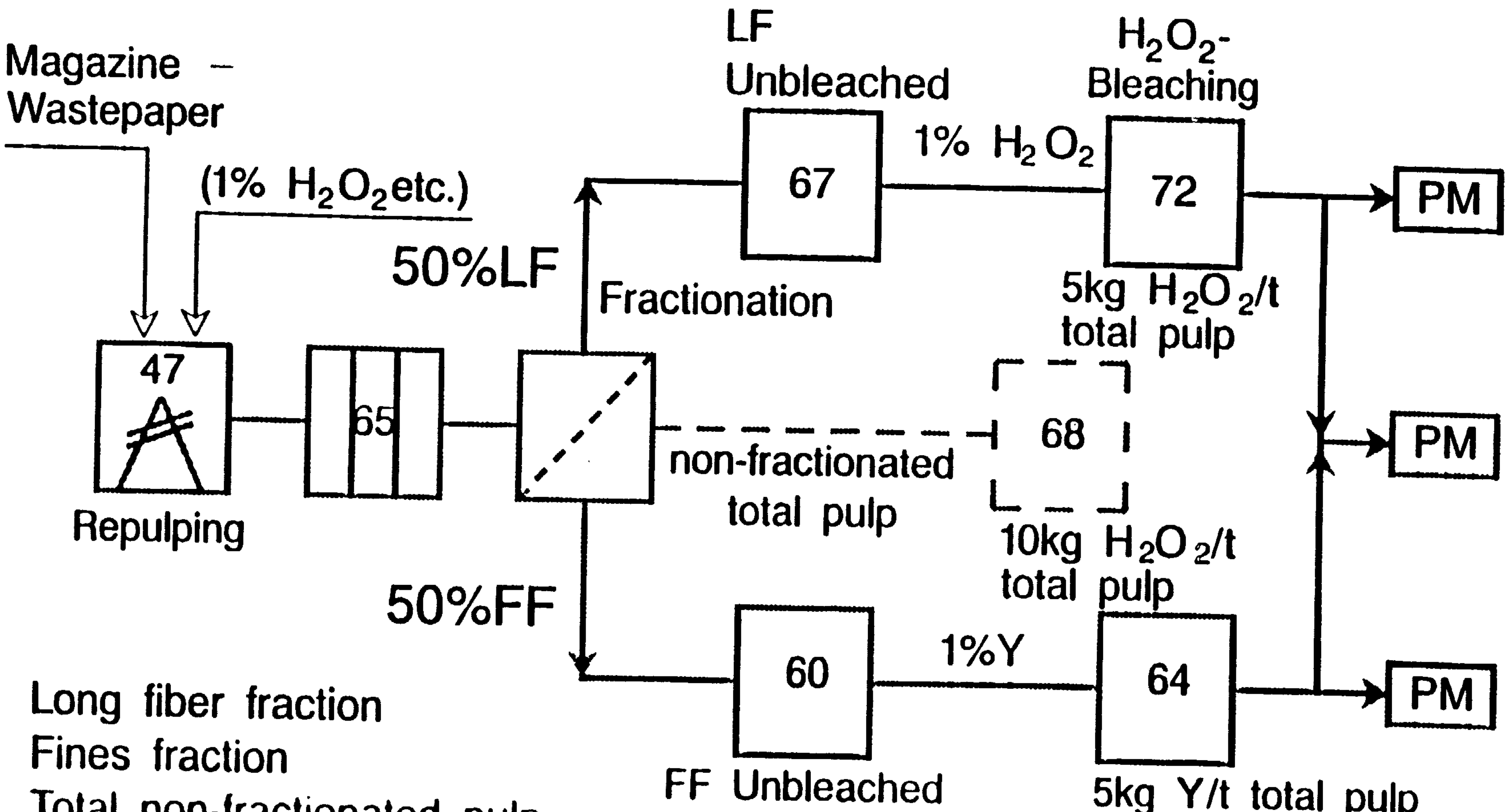
Np. = Newsprint

Mag. = Magazines

Brightness

Brightness gain (related to raw material)

Figure 3 **DEINKING PROCESS WITH POST DEINKING**



LF = Long fiber fraction
FF = Fines fraction
— = Total non-fractionated pulp
Y = Sodium dithionite
PM = Paper machine

Figure 4

DEINKING PROCESS WITH FIBER FRACTIONATION

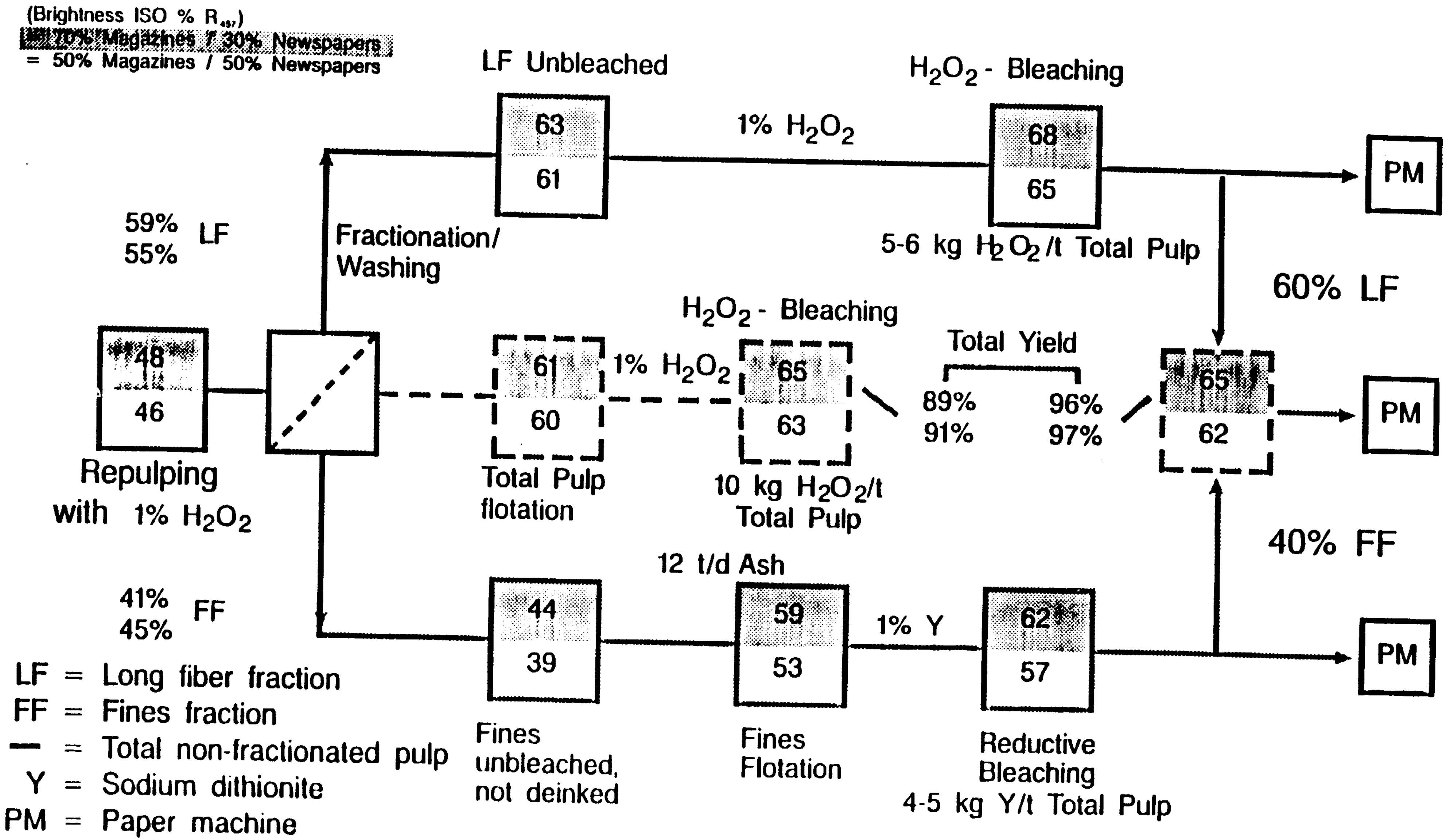
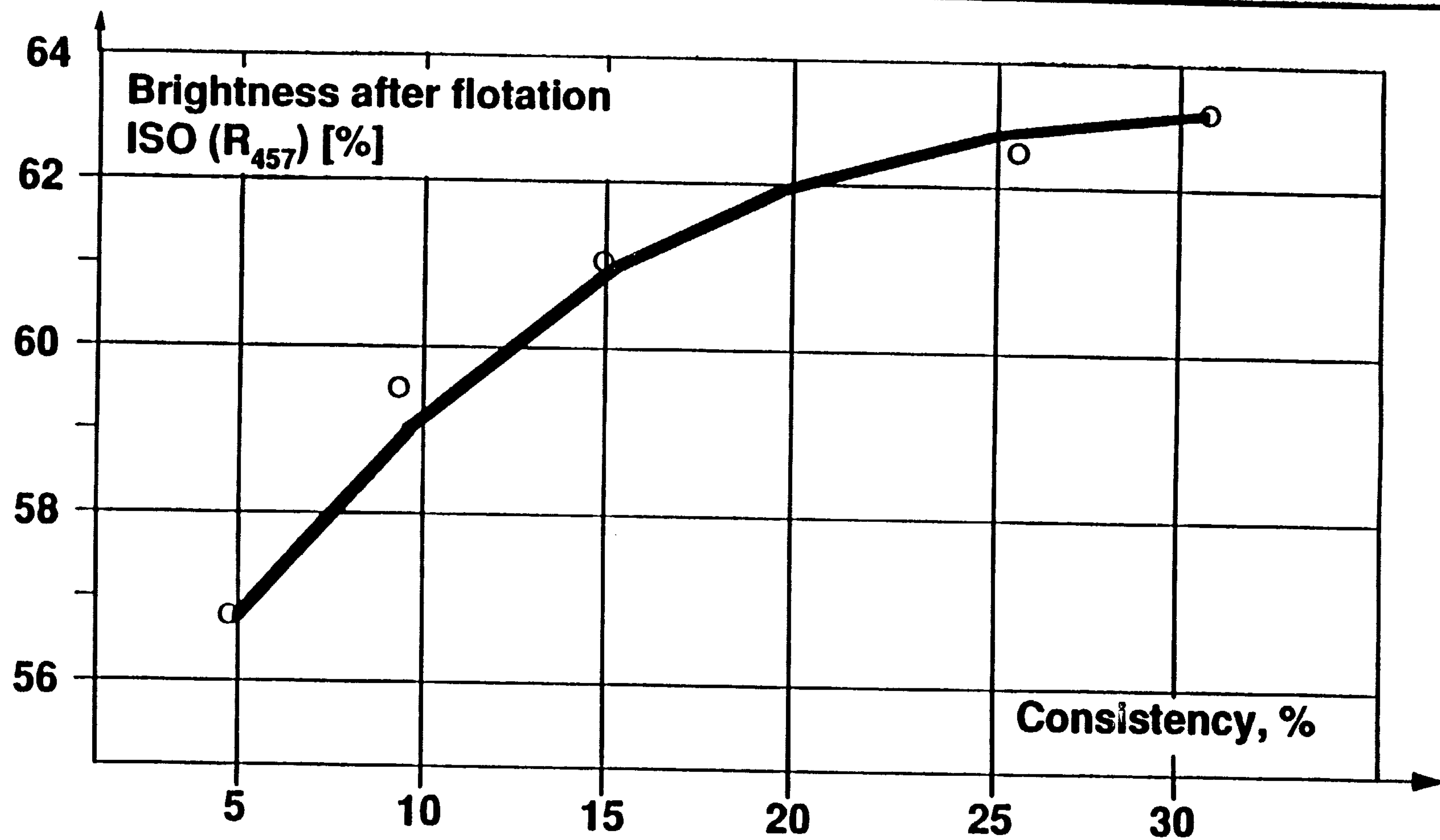


Figure 5

DEINKING PROCESS WITH FIBER FRACTIONATION PRIOR TO FLOTATION



Chemicals: 1% H_2O_2 etc.

Wastepaper: 50% Newsprint / 50% Magazines

Figure 6 THE EFFECT OF STOCK CONSISTENCY DURING REPULPING UNDER STANDARD CONDITIONS

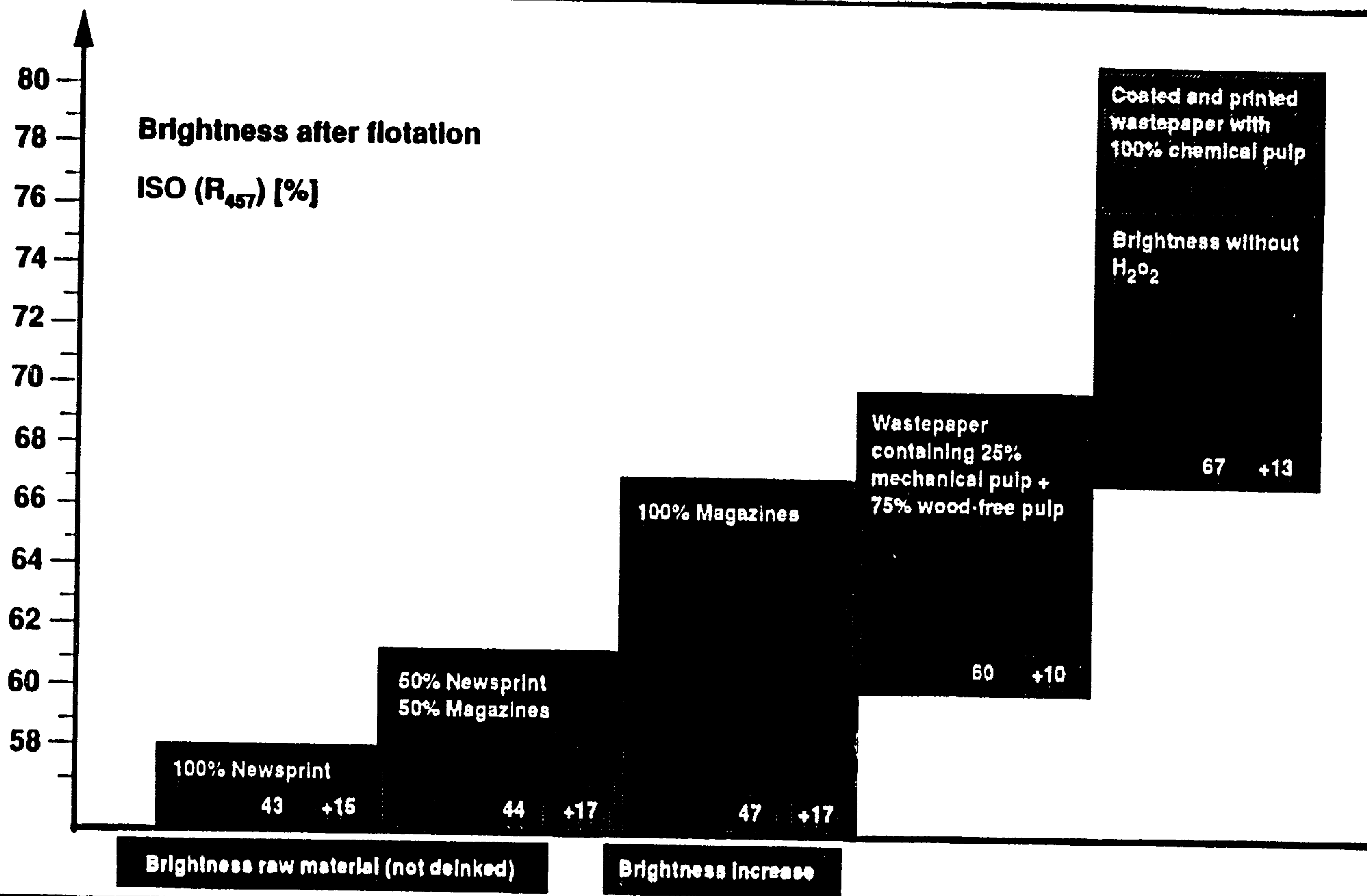


Figure 7

THE EFFECT OF 1.0% H_2O_2 IN THE PULPER ON BRIGHTNESS OF VARIOUS WASTEPAPER GRADES

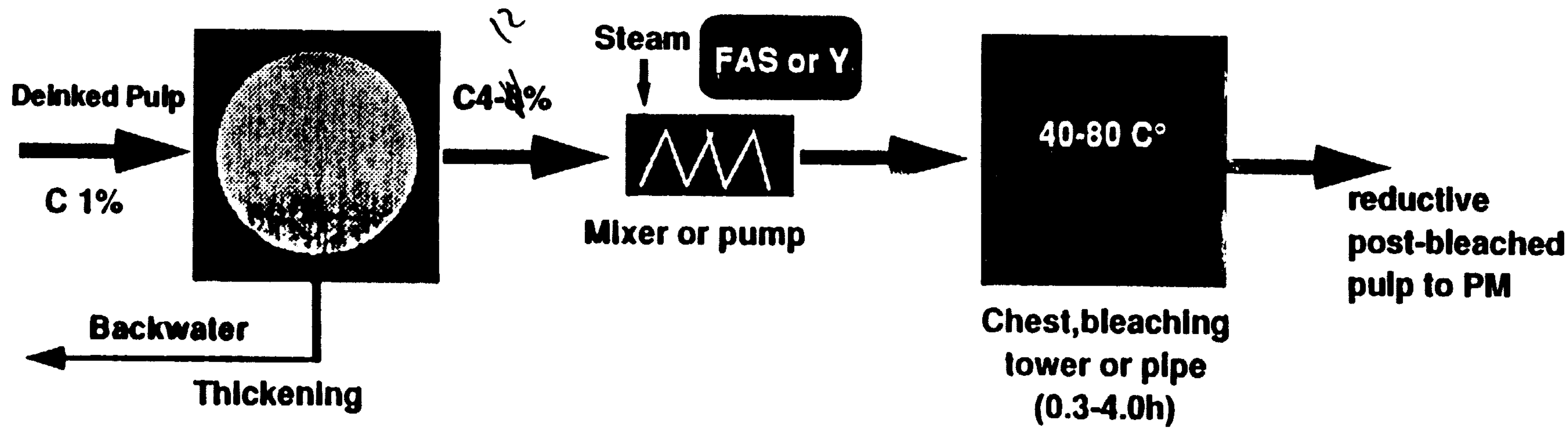


FIGURE 8

REDUCTIVE POST-BLEACHING WITH FAS OR SODIUM HYDROSULFITE

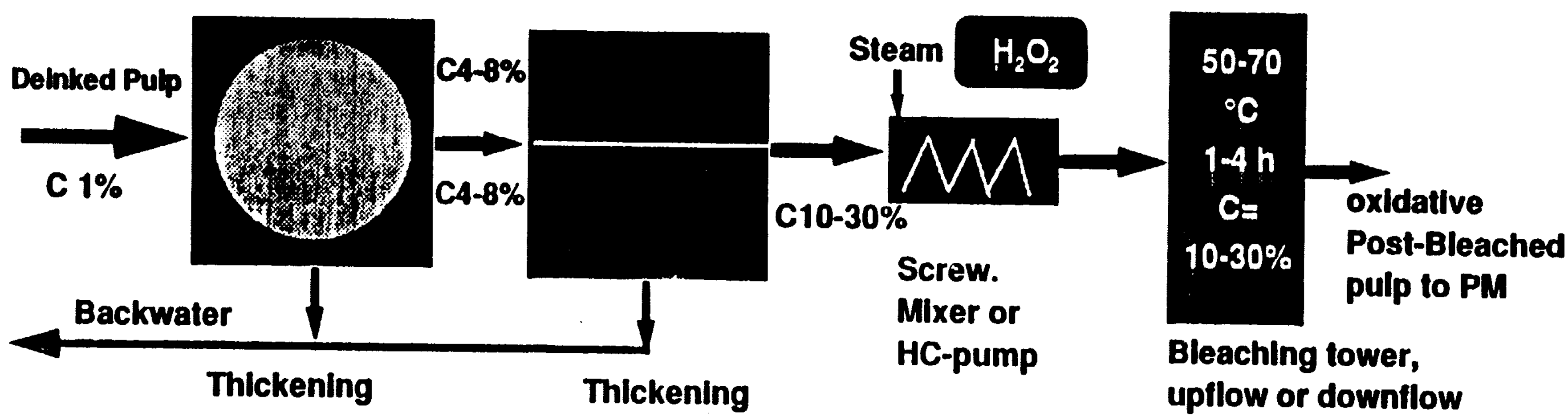


Figure 9 OXIDATIVE POST-BLEACHING WITH H_2O_2

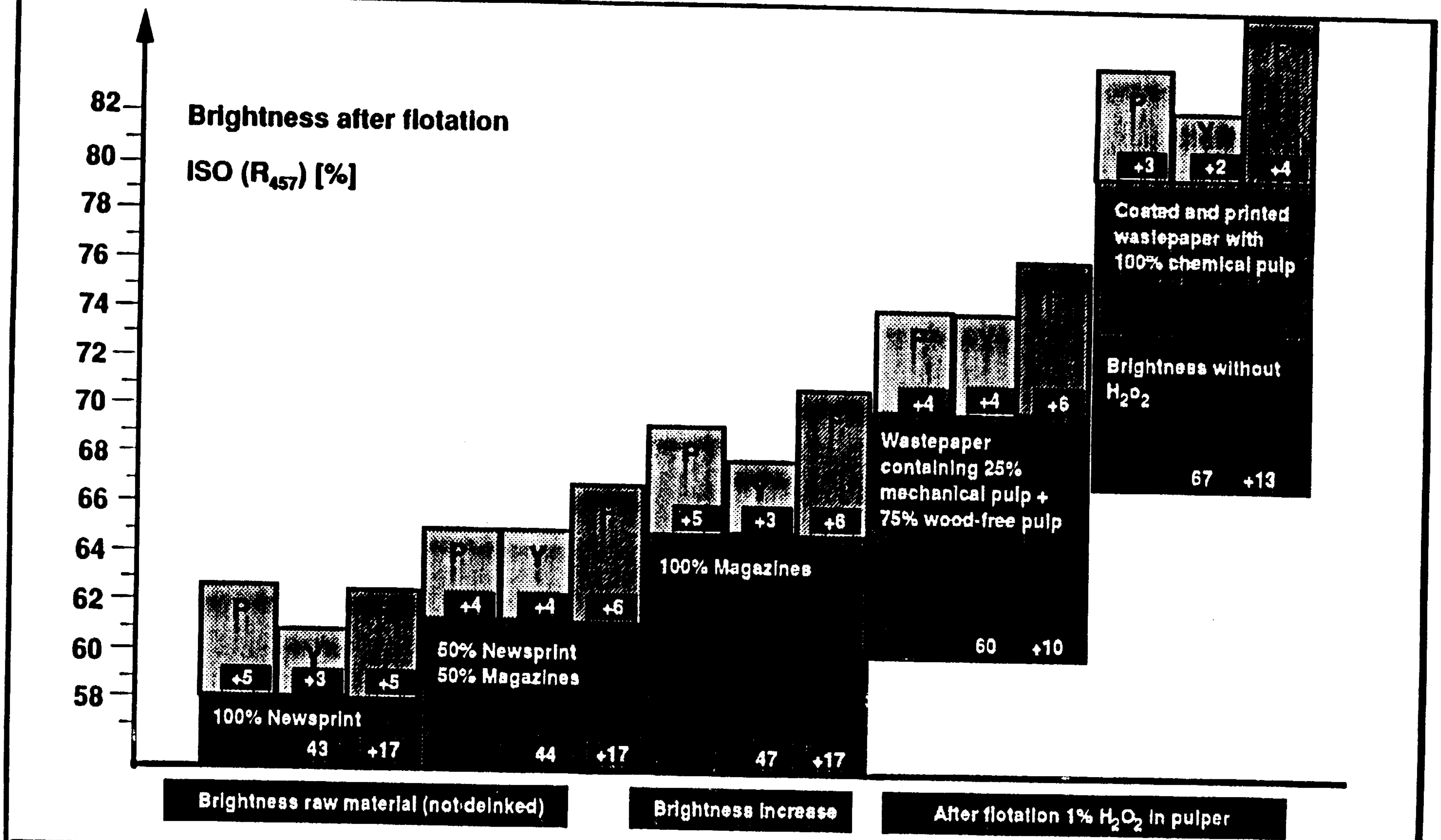


Figure 10

POST-BLEACHING OF DEINKED PULPS WITH H_2O_2, Y, FAS

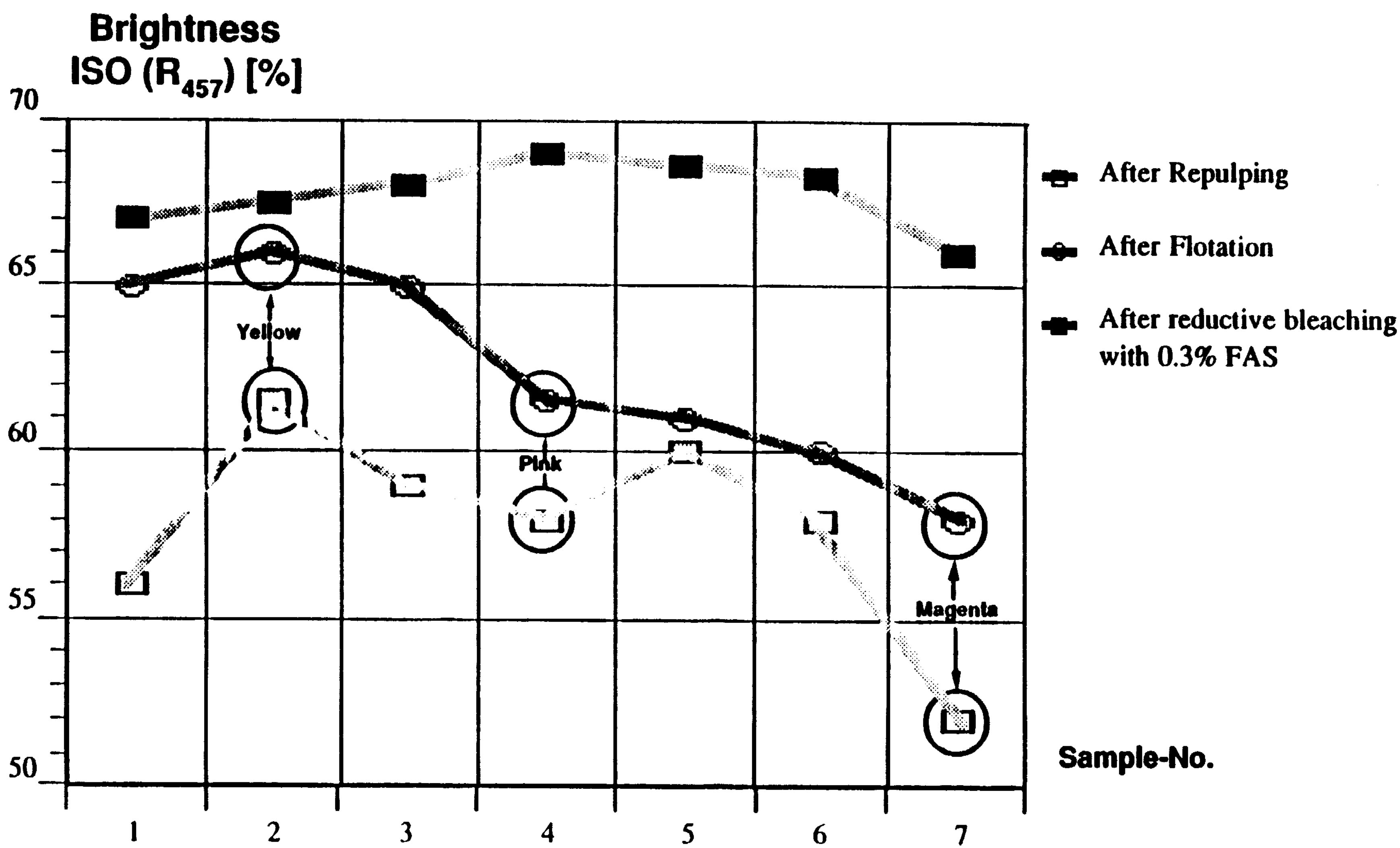


Figure 11

POST-BLEACHING OF PARTIALLY MASSDYED DEINKED STOCK 0-3% FAS

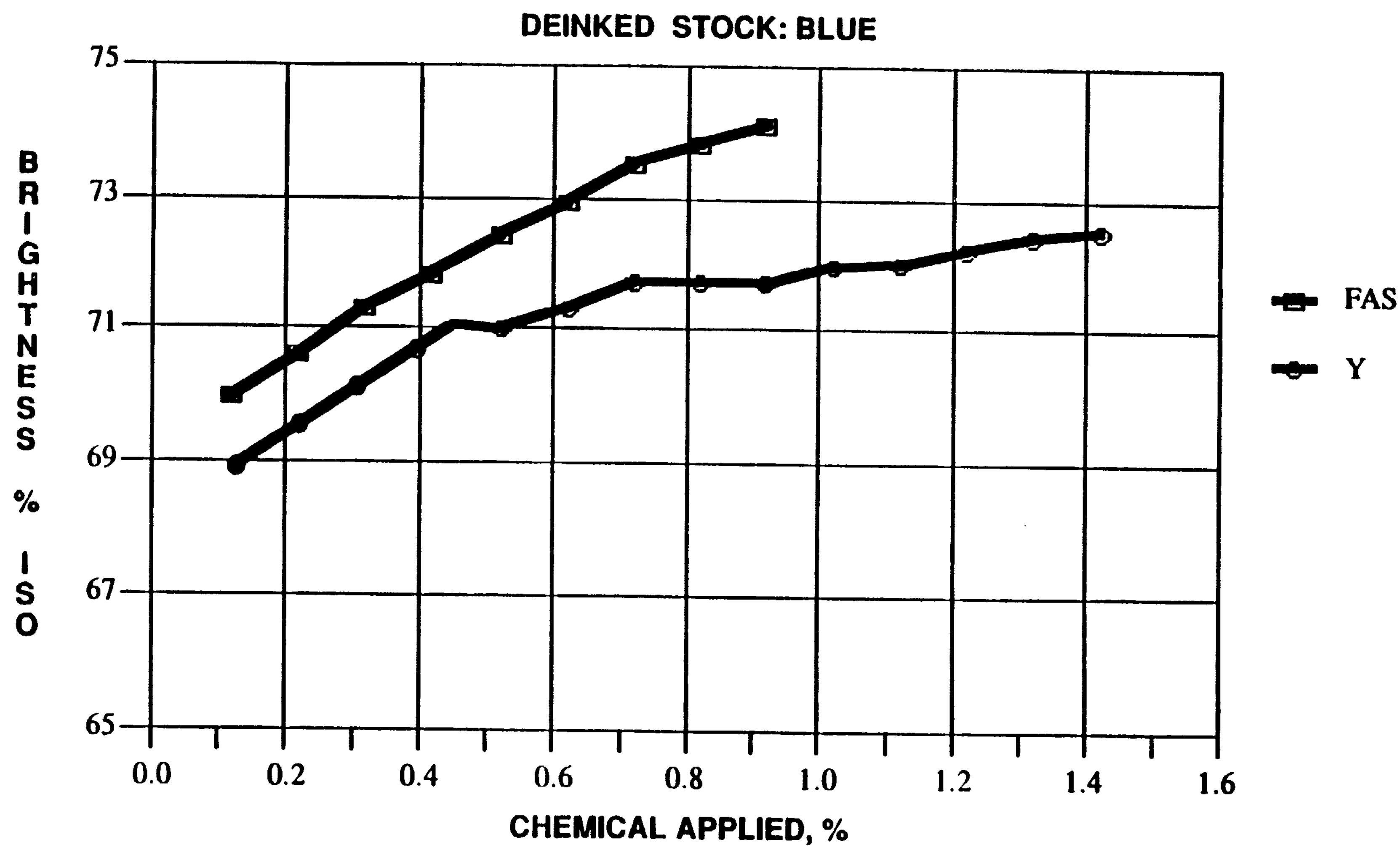


Figure 12

THE EFFECT OF FAS AND Y ON BLEACHING BLUE DEINKED STOCK

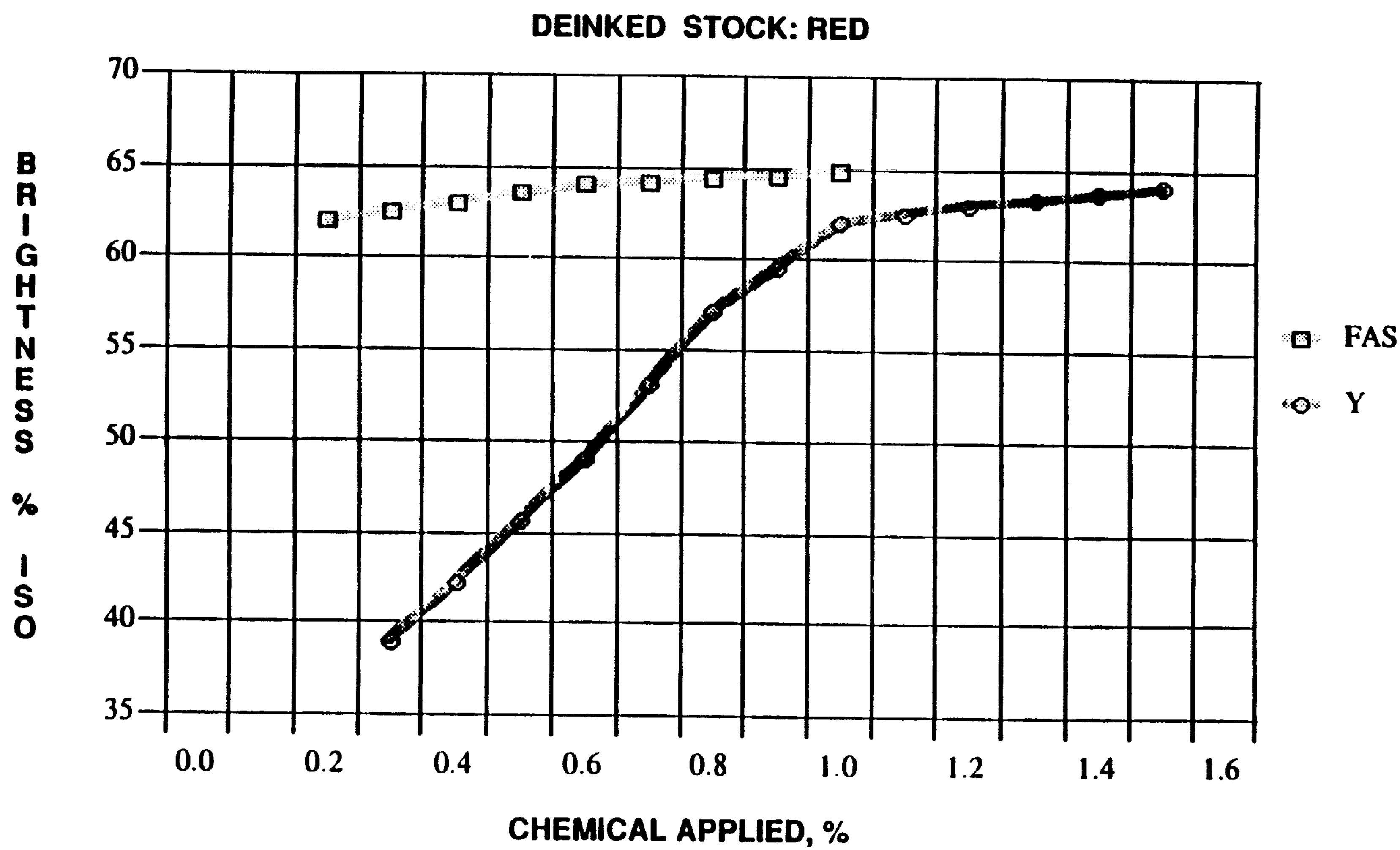


Figure 13

THE EFFECT OF FAS AND Y ON BLEACHING RED DEINKED STOCK

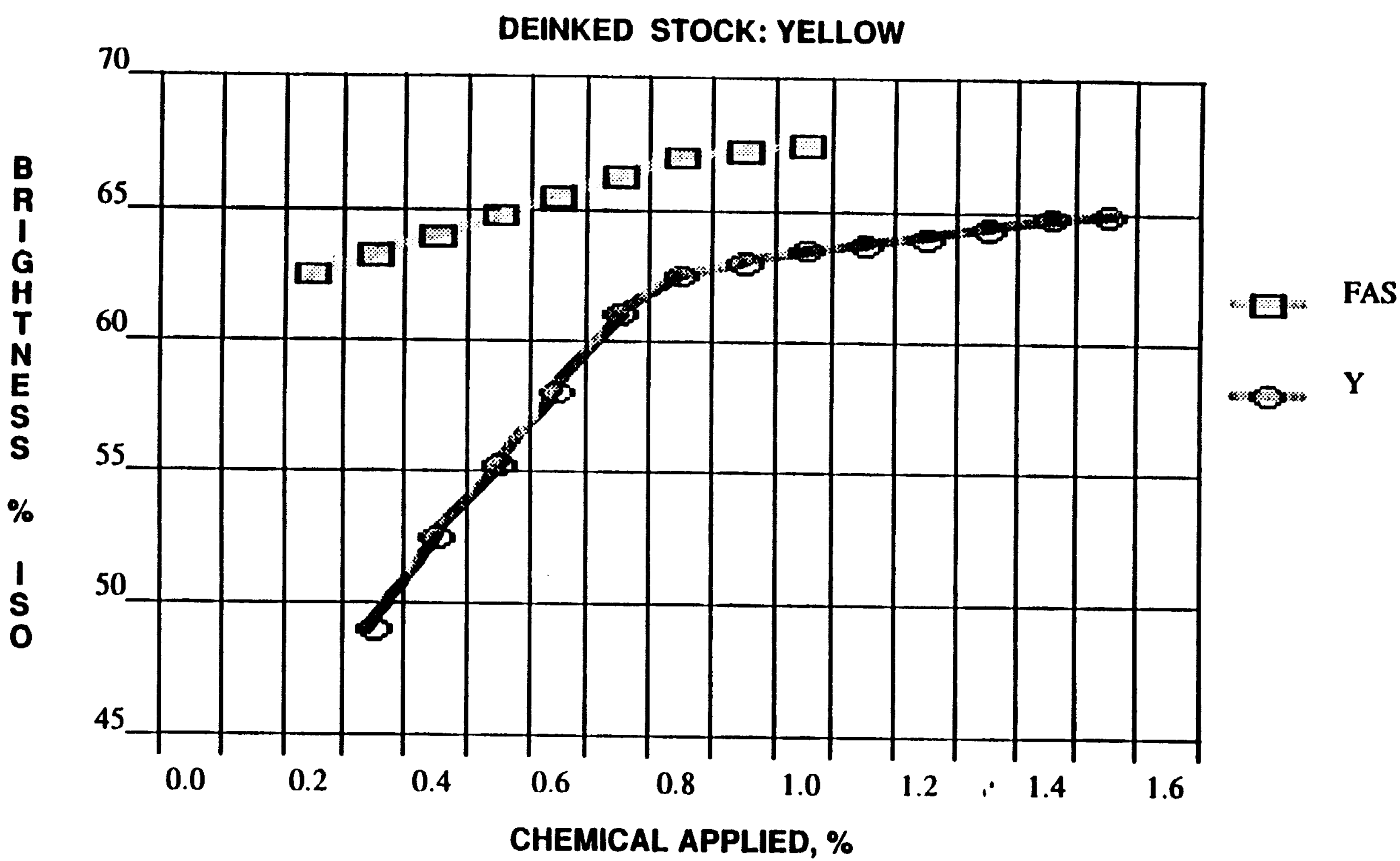


Figure 14

THE EFFECT OF FAS AND Y ON BLEACHING YELLOW DEINKED STOCK

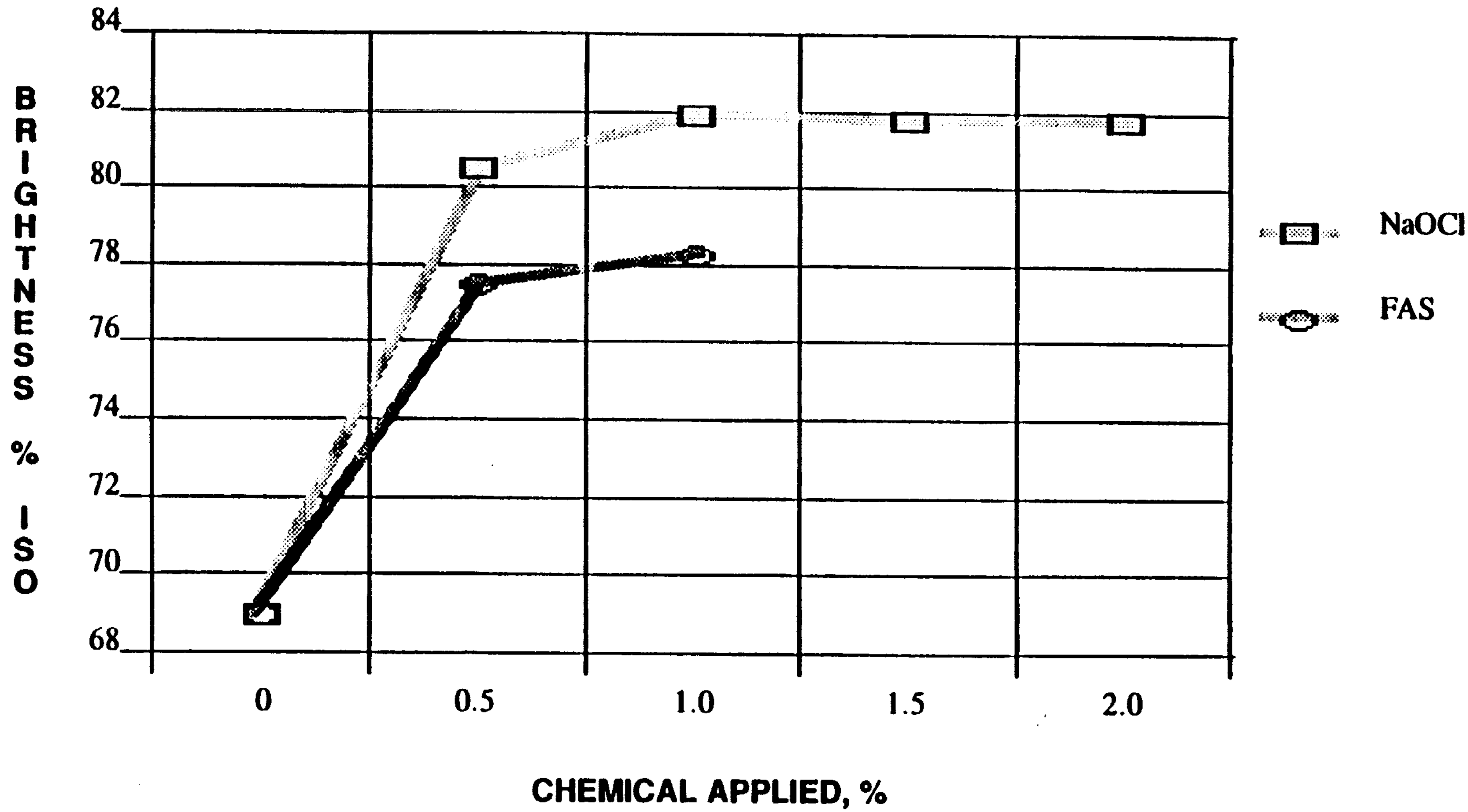


Figure 15

POST-BLEACHING OF 75% MAGAZINES DEINKED STOCK WITH FAS AND NaOCl

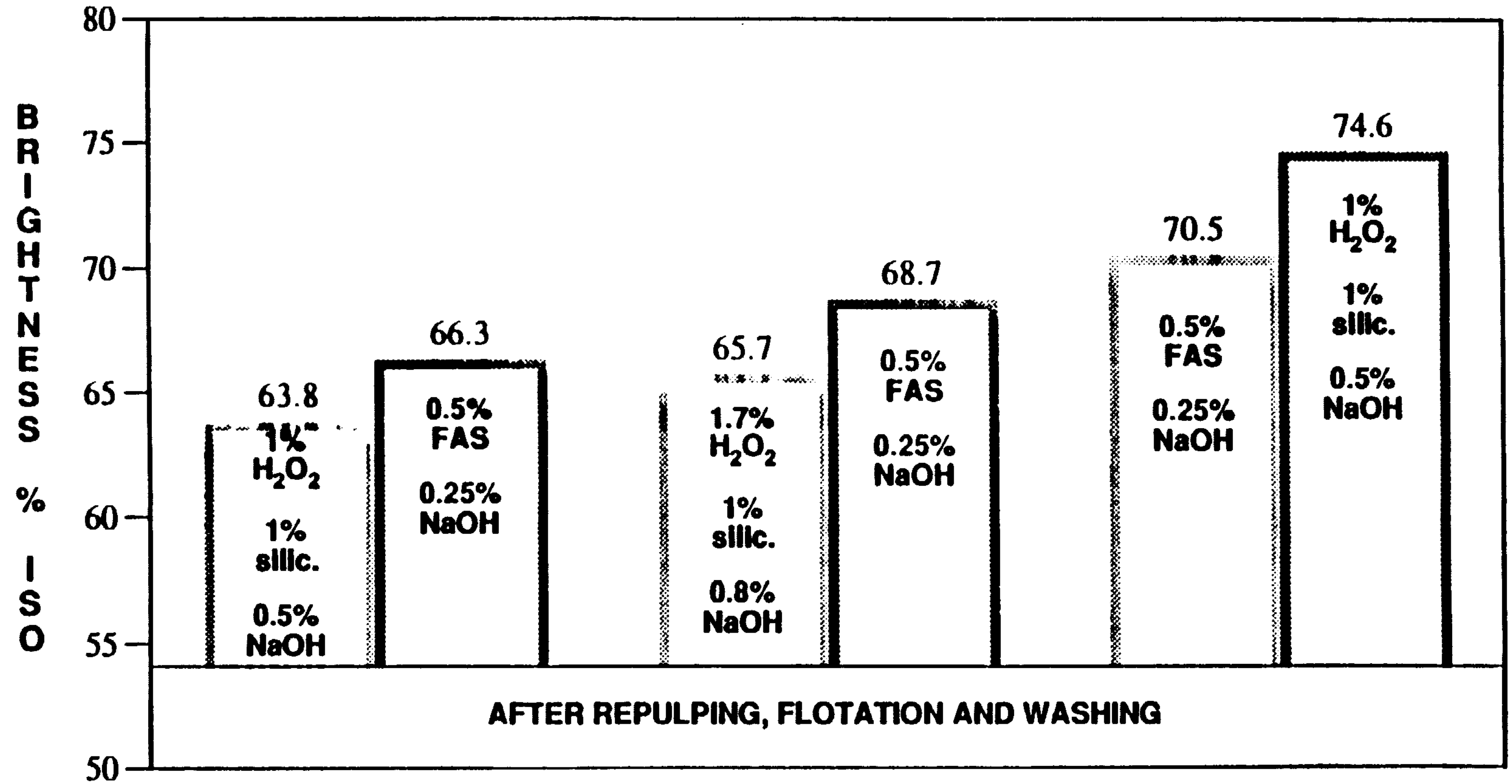


Figure 16 **TWO STAGE P-FAS AND FAS-P BLEACHING**

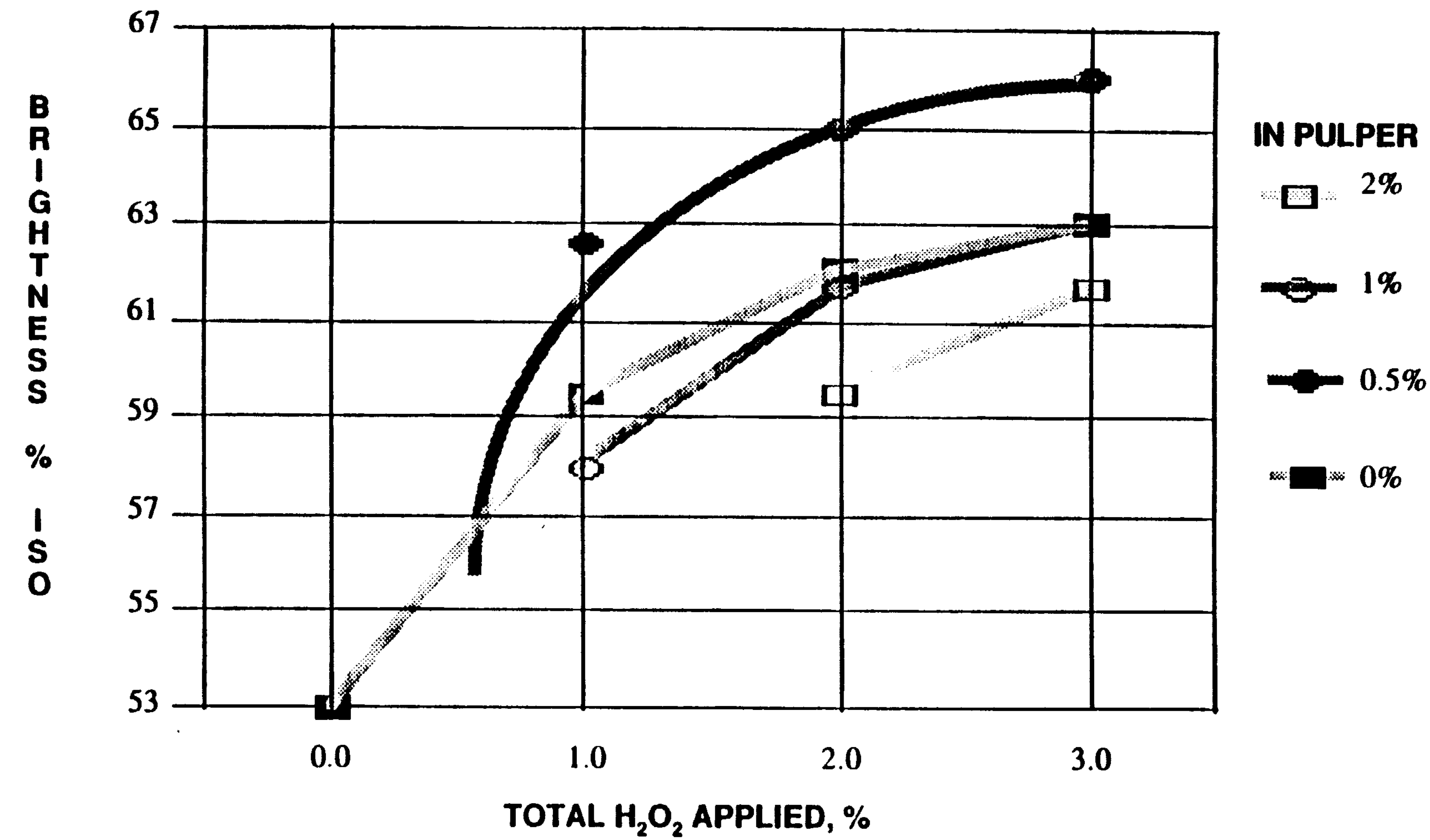


Figure 17

THE EFFECT OF SPLIT H_2O_2 ADDITION ON 75% NEWSPRINT

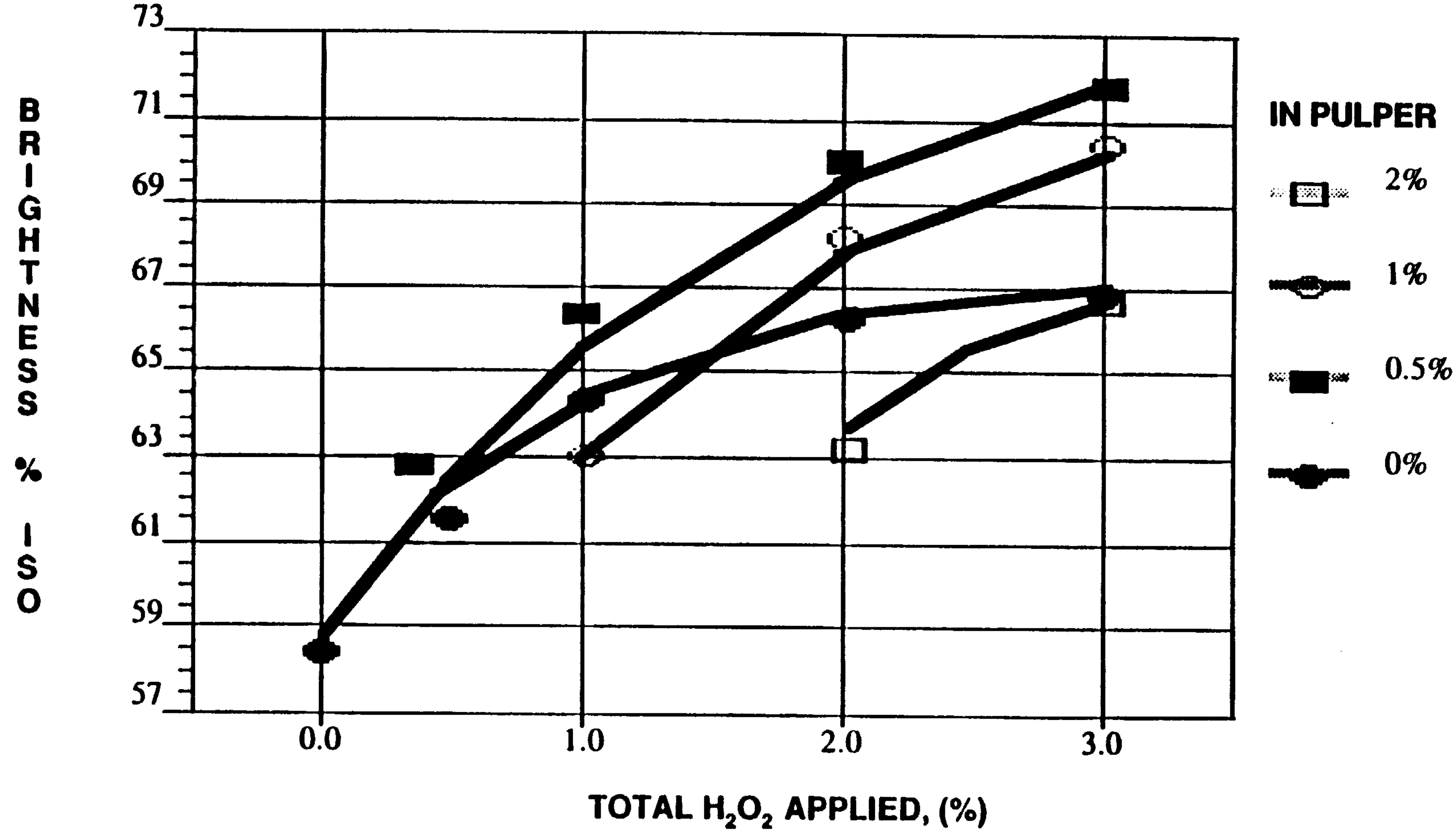


Figure 18

THE EFFECT OF SPLIT H_2O_2 ADDITION ON 75% MAGAZINES

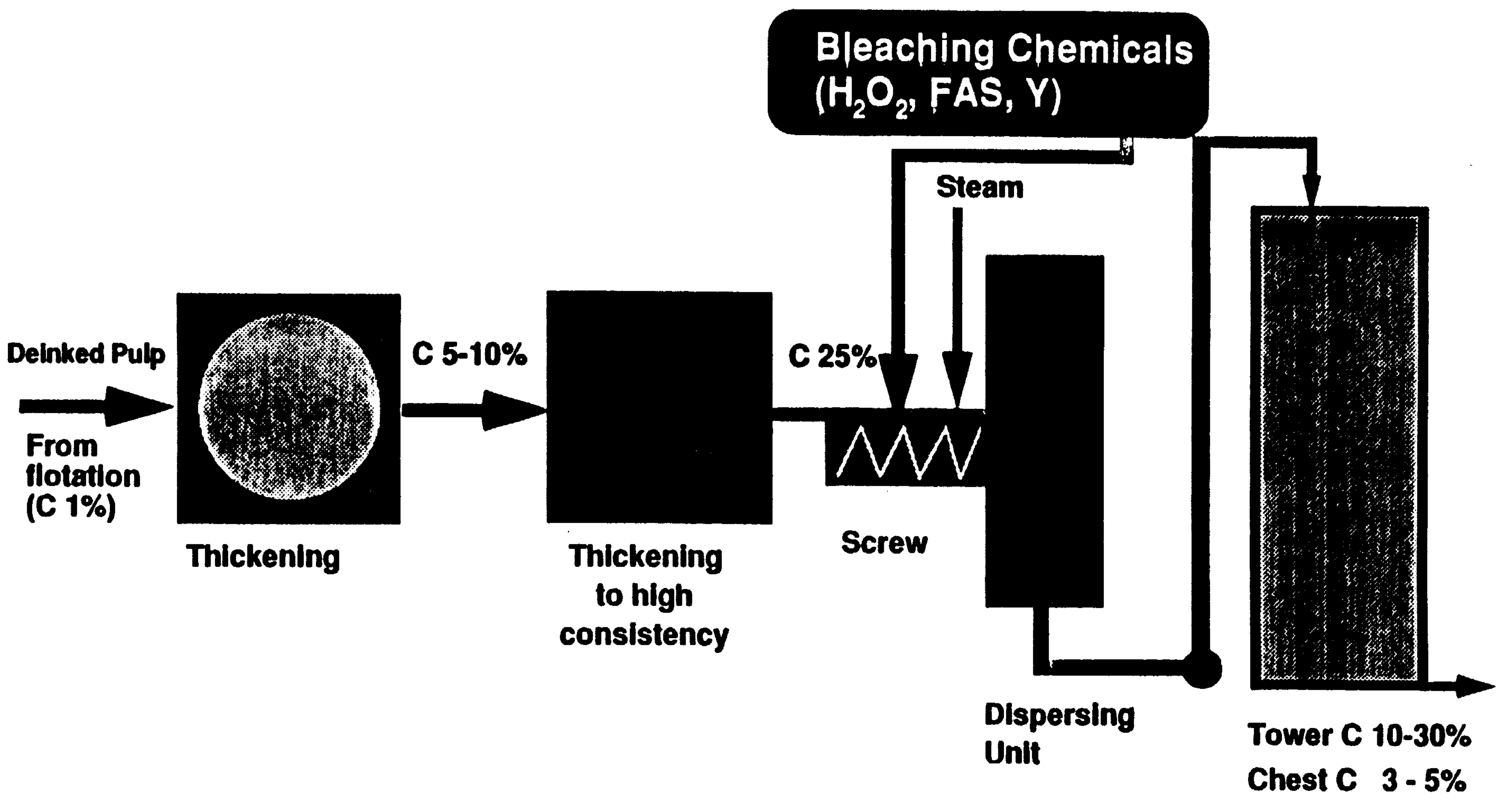


Figure 19 APPLICATION OF CHEMICALS FOR DISPERSER BLEACHING

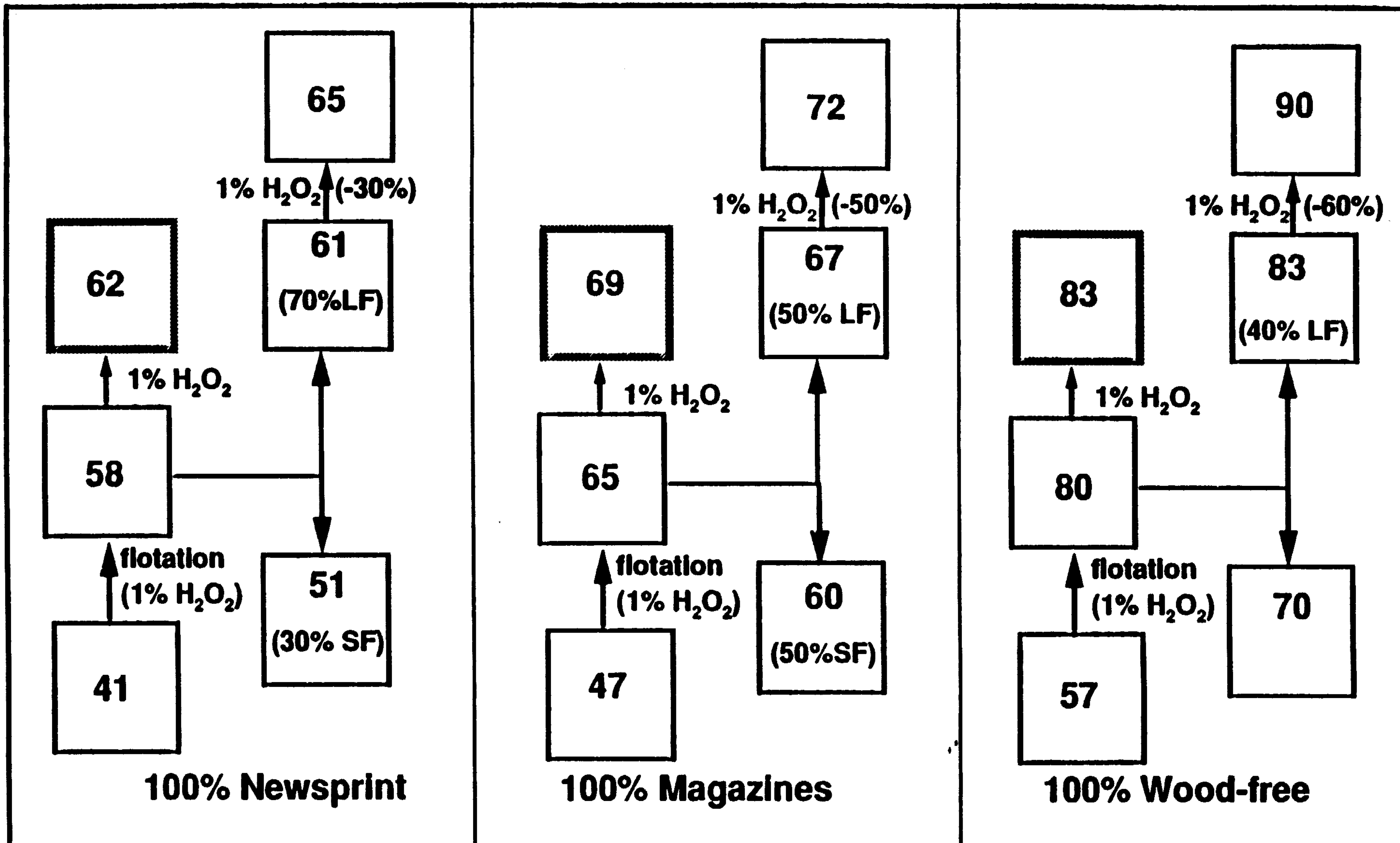


Figure 20 THE EFFECT ON POST-BLEACHING AFTER FIBER FRACTIONATION ON BRIGHTNESS AND BLEACH CHEMICAL SAVING

Interoffice Memo

Degussa

Degussa Corporation

(3)

October 4, 1994

RECEIVED

95 APR -4 PM 12:02

TO: Mike Berger cc: Robert Marion

FROM: Terry W. Maples

RE: **MEMO TO FIELD SALES CONCERNING FAS**

After reviewing the new health hazard information for FAS, minor revisions have been made to the MSDS (see noted revisions). It is recommended that the following text be sent to all field sales personnel in a memo which precedes the revised FAS MSDS:

> Preliminary and unpublished toxicology studies conducted in Austria have shown high doses of FAS to cause mutagenic effects in animals. Mutagenic substances can cause changes in cells which could result in adverse reproductive effects. Degussa is attempting to review this study.
>
> If the study is published without revisions, the material safety data sheet (MSDS) will be revised to reflect this new information. The current MSDS adequately addresses the necessary exposure controls; however, the MSDS has been revised to put a stronger emphasis on ventilation controls to eliminate airborne dust at the source point.
>
> There are no present federal, state or local regulatory requirements that affect the handling or use of mutagenic substances as a category of chemicals. As with all chemicals, appropriate exposure controls and proper handling are required to maintain employee exposures at safe levels.
>
> Degussa Corporation, as a member of the Chemical Manufacturers Association (CMA), operates under the guiding principles of Responsible Care® "to extend knowledge by conducting or supporting research on the health, safety, and environmental effects of our products."

Since some of our customers may already have knowledge of this study, you should provide them with this clarification and Degussa's position during your customer contacts.

Degussa AG toxicologist, Dr. Mayr, has been contacted and is evaluating this entire issue. Dr. Mayr will advise DC very soon of DAG's position on this study. If you require additional information or assistance, please call.

Terry W. Maples

Terry W. Maples, CIH
Manager Corporate Industrial Hygiene

Interoffice Memo

Degussa

Degussa Corporation

(4)

RECEIVED

95 APR -4 PM 12:02

October 4, 1994

TO: SALES FORCE

CC: M. DYE
J. KRONIS
R. MARION
A. NEDZA
R. WARDELL

FROM: [redacted] Mike

RE: FAS MSDS REVISION

After reviewing the new health hazard information for FAS, minor revisions have been made to the MSDS (see noted revisions). It is strongly recommended that the following information be explained to your FAS customers during a personal visit as soon as possible. A revised MSDS should be delivered to the customer at that time.

Since some of our customers may already have knowledge of this study, it is important to provide them with this clarification and Degussa's position during your customer visit.

1. Preliminary and unpublished toxicology studies conducted in Austria have shown high doses of FAS to cause mutagenic effects in animals. Mutagenic substances can cause changes in cells which could result in adverse reproductive effects. Degussa is attempting to review this study.

2. If the study is published without revisions, the material safety data sheet (MSDS) will be revised to reflect this new information. The current MSDS adequately addresses the necessary exposure controls; however, the MSDS has been revised to put a stronger emphasis on ventilation controls to eliminate airborne dust at the source point.

3. There are no present federal, state or local regulatory requirements that affect the handling or use of mutagenic substances as a category of chemicals. As with all chemicals, appropriate exposure controls and proper handling are required to maintain employee exposures at safe levels.

4. Degussa Corporation, as a member of the Chemical Manufacturers Association (CMA), operates under the guiding principles of Responsible Care® "to extend knowledge by conducting or supporting research on the health, safety, and environmental effects of our products."

Degussa AG toxicologist, Dr. Mayr, has been contacted and is evaluating this entire issue. Dr Mayr will advise DC very soon of DAG's position on this study. If you require additional information or assistance, please call Terry W. Maples, Manager Corporate Industrial Hygiene at (205) 443-4000.

Since some of our customers may already have knowledge of this study, you should provide them with this clarification and Degussa's position during your customer contacts.

M. Berger

MIB/clf/fasmsds