Paper industry *biocides*
Innovation, preservation and disinfection
BASF is one of the world’s leading suppliers of specialty biocides. We create products that make life safer, simpler and more comfortable for millions of people worldwide.

Our high performance biocides are used in every aspect of life. From toiletries, to air conditioners used in both industry and home, from Iceland to Equador our technology spans the globe in preservation and disinfection. We help to protect the lives of millions of people across the world every day.

Through our product range, marketing expertise, technical and regulatory knowledge, BASF’s innovative biocides approach positions the business at the very forefront of antimicrobial technology. The marketing, technical and research and development headquarters for BASF’s specialty biocide business is located in Nottingham.

Our products are made in ‘state of the art’ manufacturing facilities at Cramlington, England and Ludwigshafen, Germany. In each of our plants, the expert integration of process and energy requirements leads to manufacturing efficiencies and quality production.

To demonstrate our commitment to our customers, we have made significant investment in long term dedicated biocide resources within Europe, Asia Pacific and the USA.

In 2000, we established a direct presence in the USA with a new dedicated marketing and technical centre based at BASF Corporation headquarters in Mount Olive, New Jersey. Product is supplied through an integrated BASF warehousing facility in Charlotte, North Carolina. Our US operation is part of BASF Corporation’s Chemical Division, situated in Mount Olive, New Jersey.

How we work for you

BASF’s biocides are sold through BASF’s established network of sales offices worldwide. In this way, we aim to offer valuable local support and the highest standards of customer excellence.

Working with BASF provides access to many essential regulatory approvals. For example our EPA registrations allow products and customer formulations to be sold in the USA and our approvals ensure supply to the paper industry in the Nordic area. We are also well positioned to support our actives under the European Biocidal Products Directive.

We believe in long-term commitment to our products and customers and this is reflected in our substantial registrations and the data required to obtain them. To this end many of our products are supported with extensive safety and environmental data packages.

In addition, BASF is committed to providing the highest standard of health, safety and environmental protection as part of our commitment to Responsible Care. We continually research and innovate to produce the most advanced products on the market.

At BASF, we are also constantly reviewing ways in which we can improve our products and services to ensure that we keep pace with the rapidly changing developments in information technology. BASF is harnessing these changes and ensuring that we are aligned with our customer’s requirements, making it more efficient and convenient for you to do business with us.

BASF’s specialty biocide products are marketed under the Myacide® and Protectol® trade names.
Introduction to the Paper Biocide Industry

Microbiological Problems in the Paper Industry

The paper and paperboard industry is highly diverse. Products range from specialised papers for currency printing through to tissue and newsprint production and the manufacture of paperboard for packaging. Each of these areas demands different approaches in terms of the raw materials used and the various additives needed to arrive at an acceptable finished product.

All of these areas, however, depend on a basic process where the paper fibres are suspended in a large volume of recirculating water before being removed by filtration and compacted and dried to form the paper sheet. Various water-based additives are also used to improve the efficiency of the process and control the quality of the finished paper. These include flocculants, retention aids, fillers and paper coatings.

These conditions, together with the operating temperatures, which are normally above 30°C, provide the ideal environment for the growth of microorganisms, mainly bacteria. Contamination may originate from the air, fresh water, virgin and recycled pulp or waste water.

In the manufacturing process, this can result in the build up of slime at key points in the process. If uncontrolled, the slime can accumulate and eventually break away, leading to problems such as paper breaks, spray nozzle blockage and discolouration of the finished product. Problem organisms are the aerobic slime-forming bacteria such as Pseudomonas, Klebsiella, Enterobacter and Bacillus spp. Anaerobic organisms such as the Desulphovibrio spp. can also accumulate under slime layers and other deposits leading to the production of foul odours and corrosive by-products.

Water-based mill additives including mineral slurries and paper coatings are also prone to contamination and require adequate preservation to ensure that their stability is not compromised. The major spoilage organisms are aerobic bacteria such as those mentioned above but in some instances yeasts such as Candida and Saccharomyces spp. and moulds such as Aspergillus spp. can be involved. Consequently there may be a need for broad spectrum preservation in many of these additive products. Preservation of bulk pulp may also be required during unplanned plant shut-downs.

The occurrence of these contamination and spoilage problems creates a demand for antimicrobial products, which can be ideally satisfied by products from BASF’s specialty chemical biocide range.

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Myacide® AS, AS Plus and S1/S15/S30 Biocide

Myacide AS biocide is the industrial grade of 2-Bromo-2-nitropropane-1,3-diol or Bronopol. It provides highly effective antimicrobial activity for use in diverse and demanding industrial biocide applications, combining well-proven efficacy with important environmental safety characteristics. The main benefits of Myacide AS in the paper industry are its proven performance as a slimicide active ingredient in mill process water and as an effective preservative for mill additives. The ideal physical and microbiological properties of Bronopol place it in a new generation of products, which can be seen replacing older chemistries.

QUALITY
Myacide AS is an off-white to pale yellow, free flowing, crystalline solid. The product contains a minimum of 98% 2-Bromo-2-nitropropane-1,3-diol although typical purity values are significantly higher. As supplied it is stable for a minimum of 3 years when kept under good storage conditions.

Myacide AS Plus is marketed only in the USA as an EPA end use product. The technical specification and product quality are the same as Myacide AS.

Liquid Formulations
A range of commercial formulations is readily available.

Myacide S1 is a 20% (w/v) solution in dipropylene glycol monomethyl ether and water.

Myacide S15 is a 10% (w/w) solution in propylene glycol and water.

Myacide S30 is a 30% (w/w) solution in propylene glycol and water.

ANTIMICROBIAL ACTIVITY
The spoilage of paper, pulp, mineral slurries, coatings and other additives is generally due to a mixed population of bacteria and to a lesser extent fungi. Bronopol is highly effective, particularly against aerobic slime-forming bacteria such as *Pseudomonas* spp., *Klebsiella* spp., *Bacillus* spp. and the *Enterobacter* spp. The following table compares the Minimum Inhibitory Concentrations (M.I.C.) of Bronopol against six key microorganisms.

<table>
<thead>
<tr>
<th>Test Organism</th>
<th>M.I.C. (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Staphylococcus</em> spp</td>
<td>12.5 - 50</td>
</tr>
<tr>
<td><em>Bacillus</em> spp</td>
<td>12.5 - 50</td>
</tr>
<tr>
<td><em>Desulphovibrio</em> spp</td>
<td>12.5 - 50</td>
</tr>
<tr>
<td><em>Pseudomonas</em> spp</td>
<td>12.5 - 50</td>
</tr>
<tr>
<td><em>Candida</em> spp</td>
<td>400</td>
</tr>
<tr>
<td><em>Aspergillus</em> spp</td>
<td>3200</td>
</tr>
</tbody>
</table>

M.I.C. data is generated according to in-house methodology.

There have been no reported cases of resistance to Bronopol in over twenty years of use. Recent field trials have shown excellent activity against *Pseudomonas* species resistant to other commonly used biocides such as those based on isothiazolinones.

USAGE RECOMMENDATIONS

Solubility, Stability and Compatibility
Bronopol is readily water-soluble and solutions containing up to 28% w/v are possible at ambient temperature. The compound also shows a higher affinity for polar organic solvents (e.g. propylene glycol 50% w/v) which makes it very easy to use either as a liquid or a solid.

Bronopol’s stability is one of the most important factors in the paper industry for long term preservation. It shows optimum pH stability in the acidic range with excellent efficacy. At alkaline pH, Bronopol is less stable but still provides effective control. In these systems, Bronopol reaches an equilibrium with its breakdown products, which are also microbiologically active. This self-stabilisation means that preservative efficacy can be maintained over an extended shelf life even within aggressive, more alkaline matrices.
Bronopol is compatible with a range of materials used in the pulp and paper industry such as retention aids, flocculants and other biocides such as isothiazolinones. In common with other non-oxidising biocides, strong reducing agents and oxidising agents should be avoided. However, Bronopol can be used alongside oxidising biocides in systems with alternating biocide regimes.

Recommended Dose Rates

Paper Mill Process Water
Myacide AS can be dosed as a solid at a convenient point early in the recirculating system but experience has shown that best results are obtained using formulated solutions. It should be added to the wet end of the machine, the primary white water circuit, where good dispersion can take place. The levels required will depend on the cleanliness of the system, the quality of the raw material and the degree of microbial control required. A typical regime may involve initially dosing at a level of 50g Myacide AS per tonne of finished paper product, reducing to 10-20g per tonne after 2-3 weeks. When used in combination with other biocides, the concentrations of Myacide AS can be further reduced.

Bulk Pulp
To preserve bulk pulp, control foul odours and prevent general biodeterioration of stock, Myacide AS or an associated formulation may be dosed directly into the hydropulper, machine chest or stock chest. In general, a single slug dose of between 50ppm and 250ppm active should provide control for up to 3 days or longer, depending on the initial level of contamination. In situations where contamination is high, repeat dosing every 1-7 days may be required.

Paper Mill Additives
Myacide AS or an associated formulation can be used as an effective preservative for a wide range of auxiliary products. These include mineral slurries, starch solutions, rosin sizes, polymer dispersions and paper coatings. When used alone it can be dosed at 100-500ppm active based on final product volume. However, in situations where organisms tolerant or resistant to other preservatives are encountered, the performance of Myacide AS as a combination biocide is outstanding. In these situations the dose levels of each biocide can be reduced.
CASE STUDY

The following example (fig. 1) demonstrates the efficacy of Bronopol in preserving a typical paper mill additive based on calcium carbonate. The procedure used was ASTM E 723-91 at pH 7.5 with a mixed inoculum of *Pseudomonas aeruginosa*, a second (1,2-Benzisothiazolin-3-one (BIT)-resistant) *Pseudomonas* species, *Enterobacter cloacae* and *Klebsiella aerogenes*.

Myacide AS at a level of >20ppm was able to control this mixed inoculum for a period of six weeks following a single initial challenge of $10^6$ cfu/ml and a reinoculation at 21 days. BIT at levels of up to 200ppm active was unable to control the *Pseudomonas* growth.

SAFETY CHARACTERISTICS

In order to support the safe use of Bronopol, a wide range of toxicological studies have been carried out. The extensive nature of this database is particularly helpful in supporting product registrations and answering any concerns on product safety. Bronopol is non-carcinogenic, non-mutagenic, non-embryotoxic and non-teratogenic. It is also non-phototoxic.

Bronopol is biodegradable and does not accumulate in the environment. Due to its rapid photolysis and hydrolysis, Bronopol will not, under normal use conditions, cause problems to effluent treatment plants or to the environment following discharge into wastewaters.


REGISTRATIONS

**NORTH AMERICA**

**USA**

EPA - BASF's Bronopol is approved for use in paper mill pulp and process water, starch, pigments and extender slurries.

FDA 21 CFR. - Code of Federal Regulations. Listings are:

176.300 - Paper slimicides use.

175.105 - Indirect food contact use in adhesives.

176.170 - Components of paper and paperboard in contact with aqueous and fatty foods.

**CANADA**

Health Canada have approved the use of BASF's Bronopol as a slimicide and granted a letter of 'no objection' for use in paper coatings intended for food contact.

**EUROPE (EU)**

**DENMARK**

Paper slimicide registrations and meets Nordic Swan ecolabel requirements for slimicide use in printing paper.

**FINLAND**

Myacide S1 is registered as a paper slimicide.
FRANCE
Approved for food contact use as a paper slimicide by the Ministere de la Consommation.

GERMANY
BgVV - Recommendations for food-contact applications.
XIV.2f - substances added to dispersions to prevent microbial attack.
XXXVI, B V11 - paper and board, slimicides.

NETHERLANDS
Water and paper treatment registration.

SWEDEN
Myacide S1 is registered for use as a paper slimicide.
Myacide AS meets Nordic Swan labelling requirements for slimicide use in printing paper.
Protectol® GA 50 biocide is a 50% solution of 1,5-Pentanediol commonly known as Glutaraldehyde. It is very effective against a broad spectrum of bacteria and fungi common to the papermaking industry and is useful in the prevention of slime build-up in all areas of the manufacturing process. Protectol GA 50 has a rapid speed of kill, is cost effective and easy to use. In paper processing, the principal benefit of Protectol GA 50 is that it can be used as a stand-alone product.

QUALITY
Protectol GA 50 is a clear, colourless solution containing an active content of 50% Glutaraldehyde in water.

Protectol GA 50 is classed as an aldehyde which is effective against Gram positive and Gram negative bacteria and fungi.

Protectol GA 24 is a clear, colourless solution containing an active content of 24% Glutaraldehyde in water.

Both products have a shelf life of at least six months.

ANTIMICROBIAL ACTIVITY
Protectol GA 50 is extremely effective against slime forming bacteria such as Pseudomonas aeruginosa, Staphylococcus aureus, Escherichia coli and Micrococcus spp found in fouled paper-making systems. The ability of Protectol GA 50 to rapidly reduce the level of micro-organisms present in a typical paper making process is excellent.

M.I.C. data on Protectol GA 50 has been generated using in-house methodology. It should be noted that due to the influence of media constituents, the M.I.C. results are generally higher than the effective doses required in-use.

<table>
<thead>
<tr>
<th>Test Organism</th>
<th>M.I.C. (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Staphylococcus spp.</td>
<td>50</td>
</tr>
<tr>
<td>Bacillus spp.</td>
<td>1250</td>
</tr>
<tr>
<td>Desulphovibrio spp.</td>
<td>60</td>
</tr>
<tr>
<td>Pseudomonas spp.</td>
<td>150 - 250</td>
</tr>
<tr>
<td>Candida spp.</td>
<td>1250</td>
</tr>
<tr>
<td>Aspergillus spp.</td>
<td>475</td>
</tr>
</tbody>
</table>

USAGE RECOMMENDATIONS
Solubility, Stability and Compatibility
Protectol GA 50 is soluble in water and a range of polar organic solvents. This facilitates use in the vast majority of water-based systems and ensures that it can exert maximum effect at the site of contamination in the paper process. Stability is reduced above pH7 and at elevated temperatures (>40˚C).

Protectol GA 50 is compatible with a range of materials used in the paper and pulp industry such as retention aids, flocculants and other biocides. In common with other non-oxidising biocides, strong reducing and oxidising agents should be avoided.

Recommended Dose Rates
Paper Mill Process Water
Protectol GA 50 can be dosed at a convenient point early in the process, but experience has shown that the best results are achieved by adding it to the wet end of the machine, where good dispersion can take place. Convenient locations for this are the machine chest, constant head box, machine wire pit or backwater loop system. The levels of Protectol GA 50 required depend on the cleanliness of the system, the quality of the raw materials and the degree of microbial control required. A typical regime may involve initially dosing at a level of 200 - 400g of Protectol GA 50 per tonne of finished paper.
Bulk Pulp
Protectol GA 50 can be used to preserve bulk pulp and control both foul odours and general biodeterioration of stock. It may be dosed directly into the hydropulper, machine chest or stock chest. A single slug dose of between 100ppm and 500ppm is sufficient to provide control in the stock. In situations where contamination is high, repeat dosing every 1 - 7 days may be required.

Paper Mill Additives
To inhibit the growth of spoilage bacteria during the manufacture of water-based solutions, suspension concentrates and emulsions, Protectol GA 50 may be dosed at 100-500ppm based on final formulation volume.

SAFETY CHARACTERISTICS
In order to support the safe use of Glutaraldehyde, a wide range of toxicological studies has been carried out. The extensive nature of this database is particularly helpful in supporting product registrations and answering any concerns on product safety. Protectol GA 50 is toxic if swallowed or inhaled and is corrosive to the skin and eyes. It may cause skin sensitisation and has been classed as a respiratory sensitiser in the EU. Protectol GA 50 is not teratogenic or carcinogenic and is not mutagenic in animal studies. Protectol GA 50 is readily biodegradable in the environment and should not cause problems in sewage treatment plants and surface waters when used as recommended.

REGISTRATIONS

NORTH AMERICA
USA
EPA - BASF’s Glutaraldehyde is approved for use as a paper slimicide.
FDA 21, CFR - Code of Federal Regulations. Listings are:
173.320 - Chemicals for controlling microorganisms in cane-sugar and beet-sugar mills.
176.170 - Components of paper and paperboard in contact with aqueous and fatty food.
176.180 - Components of paper and paperboard in contact with dry food.
176.300 - Paper slimicide use.

CANADA
BASF’s Glutaraldehyde is registered as a technical product for use in manufacturing registered microbiocides.

EUROPE (EU)
GERMANY
BgVV - Recommendations for food-contact applications.
XXXVI, B VII - Paper and board, slimicides.
XXXVI2, IIG - Paper and board for bakeries, slimicides.
FINLAND
Protectol GA 50 and GA 24 are approved for use in paper slimicides.
SWEDEN
Protectol GA 50 is approved for use as a paper slimicide.
Protectol® DZ / Protectol® DZ P
Biocide

Protectol DZ biocide is the trade name for Dazomet, a heterocyclic compound. Within the pulp and paper industry it is used both as a preservative for mill additives and as an active slimicide to control growth in the process water. It boasts excellent antimicrobial activity. Protectol DZ is particularly active against slime-forming organisms and sulphate reducing bacteria, which can be responsible for corrosion.

The most important benefits of Dazomet in the pulp and paper industry are its rapid speed of kill and broad spectrum activity.

QUALITY
Protectol DZ is a fine granular grade product and Protectol DZ P is a powder form. Both products are off-white in colour and contain a minimum of 99% Tetrahydro-3,5-dimethyl-2H-1,3,5-thiadiazine-2-thione.

BASF’s Dazomet has a shelf life of at least 2 years. It should not be stored at temperatures above 40°C.

ANTIMICROBIAL ACTIVITY
Dazomet is extremely effective against slime-forming and spoilage organisms such as Pseudomonas spp., Klebsiella spp., Staphylococcus aureus, Aspergillus niger and Candida albicans. The Minimum Inhibitory Concentrations (M.I.C.) of Dazomet are displayed in the following table:

<table>
<thead>
<tr>
<th>Test Organisms</th>
<th>M.I.C. (ppm)</th>
<th>20°C</th>
<th>40°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Staphylococcus aureus</td>
<td>500</td>
<td>3.9</td>
<td></td>
</tr>
<tr>
<td>Escherichia coli</td>
<td>500</td>
<td>62.5</td>
<td></td>
</tr>
<tr>
<td>Proteus mirabilis</td>
<td>250</td>
<td>15.6</td>
<td></td>
</tr>
<tr>
<td>Pseudomonas aeruginosa</td>
<td>250</td>
<td>31.3</td>
<td></td>
</tr>
<tr>
<td>Candida albicans</td>
<td>500</td>
<td>7.8</td>
<td></td>
</tr>
</tbody>
</table>

M.I.C. data is generated according to in-house methodology.

USAGE RECOMMENDATIONS

Solubility, Stability and Compatibility
Dazomet has a relatively low water solubility (max 0.3% w/w) but is sufficiently soluble to act effectively in water-based systems. The solubility of the active may be increased by the use of organic solvents (e.g. Dimethylformamide, N-Methylpyrrolidone) or by formation of sodium salts at high pH. Solutions of >20% w/w are possible in 10% NaOH. It is also possible to formulate suspension concentrates containing 30% active ingredient.

Dazomet is compatible with a wide range of additives used in the paper processing industry including flocculants, retention aids, dispersants and mineral slurries.

Recommended Dose Rates
Protectol DZ is very active at low doses in paper industry biocide applications. For use as a slimicide it should be dosed at 50g to 150g per tonne of finished paper. As a preservative for bulk pulp and paper mill auxiliaries (e.g. mineral slurries) 100-500ppm Protectol DZ is recommended.

SAFETY CHARACTERISTICS
Protectol DZ has an extensive toxicity data package due to its use in the agricultural industry. As supplied, Protectol DZ has a moderate acute oral toxicity and is an eye irritant but is neither irritant nor sensitising to the skin. Protectol DZ is also not carcinogenic, mutagenic or teratogenic. Protectol DZ is ultimately biodegradable in the environment and should not cause problems in sewage treatment plants and surface waters when used as directed.

REGISTRATIONS
NORTH AMERICA
USA
EPA - BASF’s Dazomet is approved for the manufacture of antimicrobial products. It also has end use approvals for paper slimicide applications and mill additive preservation.

FDA 21 CFR - Code of Federal Regulations. Listings are:
176.170 Components of paper and paperboard in contact with aqueous and fatty food.
176.180 Components of paper and paperboard in contact with dry food.
176.230 Preservative used in paper coatings for use in contact with food.
176.300 Paper slimicide use.

CANADA
BASF’s Dazomet is approved by Health Canada for the manufacture of formulated products.

EUROPE (EU)

GERMANY
BfV recommendations for food-contact applications.
XIV, 2f - Substances added to dispersions to prevent microbial attack.
XXXVI, B VII Paper and board, slimicides.
XXXVI/2, II G - Paper and board for bakery applications, slimicides.

FINLAND
Products based on Tetrahydro-3,5-dimethyl-2H-1,3,5-thiadiazine-2-thione are provisionally approved for use as slimicides in papermaking.

Dazomet is on the chemical register of Japan (MITI), Australia (AICS), Korea and Philippines.
Protectol® HT
Biocide

Protectol HT biocide is a 76% solution of Hexahydrotriazine, a formaldehyde-release biocide and is particularly suitable for use as a preservative in mineral slurries. It can be used as a stand-alone preservative or in conjunction with other biocides. Protectol HT is a premium quality Triazine, due to its purity, odour, stability and colour.

QUALITY
Protectol HT is supplied as a 76% solution of 1,3,5-Tris-(2-hydroxyethyl)-1,3,5-hexahydrotriazine. It is a clear, colourless to slightly yellowish solution.

As a concentrate, Protectol HT is stable for up to 12 months when stored in sealed containers at temperatures up to 30°C.

ANTIMICROBIAL ACTIVITY
The spoilage of mineral slurries, coatings and other additives is generally due to a mixed population of bacteria. The main types of microorganism found in slurries include species of the following genera: Pseudomonas, Alcaligenes, Aeromonas, Bacillus, Klebsiella, Serratia and Erwinia. The Minimum Inhibitory Concentration (M.I.C.) against a range of test organisms are displayed in the following table:

M.I.C. data is generated according to in-house methodology.

<table>
<thead>
<tr>
<th>Test Organism</th>
<th>M.I.C. (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Staphylococcus aureus</td>
<td>1000</td>
</tr>
<tr>
<td>Escherichia coli</td>
<td>1000</td>
</tr>
<tr>
<td>Proteus mirabilis</td>
<td>1000</td>
</tr>
<tr>
<td>Pseudomonas aeruginosa</td>
<td>1000</td>
</tr>
<tr>
<td>Candida albicans</td>
<td>2500</td>
</tr>
</tbody>
</table>

The widespread use of Protectol HT in preservation application areas suggests that it is compatible with a broad range of materials including mineral slurries and paper coating components.

Recommended Dose Rates
To inhibit the growth of spoilage bacteria during the manufacture, storage and distribution of water-based solutions, suspension concentrates and emulsions, Protectol HT may be dosed at between 250 - 1000ppm based on final formulation volume.

CASE STUDY
An in-house study was conducted to demonstrate the efficacy of Protectol HT in the preservation of calcium carbonate slurries. The procedure used was the ASTM: E723-91 Standard Test Method for efficacy of antimicrobials as preservatives for aqueous-based products. The pH of calcium carbonate clay slurry is normally between 9.0 - 10.0. In this study, Protectol HT provided long term preservation by reducing contamination to <10cfu within 24 hours and controlled microbial growth for at least 6 weeks. As a result of these studies, it is recommended that Protectol HT can be used for the preservation of calcium carbonate clay slurries at concentrations between 250 and 1000ppm.

SAFETY CHARACTERISTICS
As a concentrate, Protectol HT has moderate acute oral toxicity and is an eye irritant. It may cause skin sensitisation but it is not mutagenic.

Protectol HT is readily biodegradable in the environment and should not cause problems in sewage treatment plants and surface water when used as recommended.

REGISTRATIONS

USA
TSCA listed.

JAPAN
MITI listed.
The information submitted in this publication is based on our current knowledge and experience.

In view of the many factors that may affect processing and application, this data does not relieve processors of the responsibility of carrying out their own tests and experiments; neither do they imply any legally binding assurance of certain properties or of suitability for a specific purpose. It is the responsibility of those to whom we supply our products to ensure that any proprietary rights and existing laws and legislation are observed.