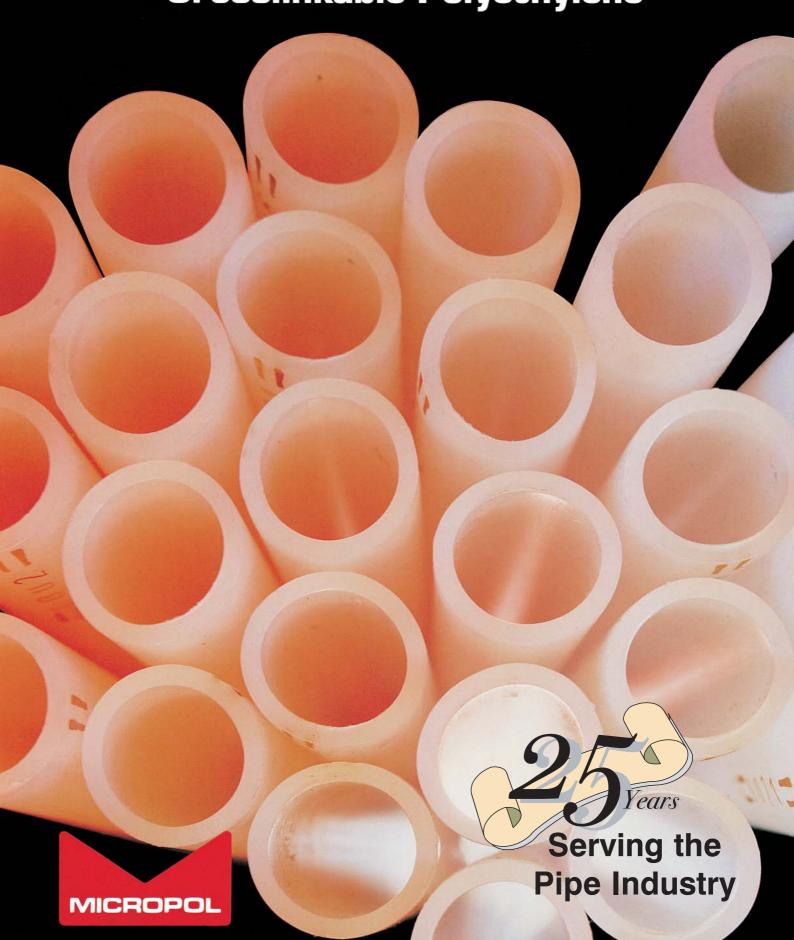
ISOPLAS Crosslinkable Polyethylene



ISOPLAS CROSSLINKABLE POLYETHYLENE

ISOPLAS crosslinkable polyethylene is produced by grafting of organo-silanes on to polyethylene.

MICROPOL'S development of this technology has created a range of easily processable polyethylenes that have outstanding resistance to rigorous environments caused by:-

LARGE TEMPERATURE CHANGES e INCREASED PRESSURE
e INCREASED PHYSICAL LOADS e EXPOSURE TO ULTRA VIOLET LIGHT
e CONTACT WITH AGGRESSIVE CHEMICALS e AND ABOVE ALL EXCEPTIONAL
RESISTANCE TO COMBINATIONS OF ALL OR SOME OF THESE RIGOROUS
ENVIRONMENTS WHERE CONVENTIONAL THERMOPLASTIC MATERIALS HAVE
PREVIOUSLY FAILED

The place of ISOPLAS crosslinkable polyethylene in the field of thermoplastic materials can best be seen by comparing it briefly with other thermoplastics and other types of crosslinkable polyethylenes.

All thermoplastic materials, such as PVC, polypropylene, nylon, polycarbonate, polybutylene, low and high density polyethylene, soften and finally flow at elevated temperatures. Crosslinking of polyethylene prevents this thermal flow by converting a thermoplastic material into a thermoelastic one. This can be visualised as the joining together of the long chain of polymer molecules with covalent bonds. This interlinking of the polymer structure significantly enhances its properties over a range of conditions.

ISOPLAS crosslinkable polymer supplied by Micropol has two components:- silane grafted polyethylene (VPE-b or **Graft Copolymer**) and **Catalyst Masterbatch**. These are supplied as granules which are blended together (normally by dosing equipment) and simply fed into the pipe extruder, moulding machine or other polyethylene processing equipment for converting to finished products. No crosslinking takes place at this stage which means that the full range of conventional process equipment and conditions can be used, without the limitations imposed by other crosslinking processes or the need for expensive machine modifications.

The finished product (which is still thermoplastic at this stage) can now be chemically crosslinked by reacting it with hot water or steam baths or saunas in a separate process. ISOPLAS crosslinkable polyethylenes have been established for twenty five years for the production of tubes for central heating, domestic hot and cold water and underfloor heating systems. The heating industry has exploited the outstanding high temperature physical properties and long service life of our grade range in many different pipe constructions.

Other industries including gas, oil and water supply, automotive, plumbing fittings, pipe insulations and refurbishment, building laminates and tie layer structures are now starting to utilise the other performance benefits of ISOPLAS

The ISOPLAS grade range is detailed on page 4 of this brochure. As well as the original range developed for the pipe extrusion industry, many modified formulations have been developed for other industries and applications. The Micropol Technical Services Department will be pleased to answer any enquiries for specialist applications. An ISOPLAS MANUAL is available which advises on the use and applications of ISOPLAS as well as detailing sources of process machinery, testing, applications, approvals, co-extrusion materials etc.

CHEMISTRY

Polyfunctional organo-silanes, containing unsaturated Vinyl groups with easily hydrolysable alkoxy functionality are chemically grafted onto the polyethylene backbone according to the following reaction.

After grafting the material is still thermoplastic and can be processed in the same way as normal (non-crosslinkable) polyethylene. Crosslinking of this grafted material is subsequently induced by exposure to trace amounts of water at elevated temperatures which cause hydrolysis and condensation of the alkoxy groups to form siloxane crosslinks.

This crosslinking reaction is normally accelerated by the incorporation of a catalyst.

These diagrams imply a polyfunctional character for the grafted silane with three alkoxy groups present at each silicon atom. Therefore, the grafted polyethylene chain is capable of reacting with two or more similar chains to form a "bunch like" crosslink structure.

The behaviour of this three dimensional crosslinked network structure is significantly different to that of the planar structure of the peroxide and radiation crosslinked polyethylenes. Hence to achieve the same low hot elongation and low deformation under load, the degree of crosslinking of peroxide crosslinked p.e. has to be 15-20% greater than that of ISOPLAS.

We can also conclude that the "bunch like" crosslink structure of ISOPLAS will give superior high temperature creep performance compared to other crosslinking techniques or the molecular entanglement employed by thermoplastic alternatives such as polybutylene, linear p.e. or polypropylene.

MANUFACTURING PROCESS

The two components that make up ISOPLAS crosslinkable polyethylene are made on compounding equipment specially designed to provide optimum mixing and control of processing conditions. Purpose built reciprocating and twin screw extruders achieve the high quality dispersions vital for handling the sensitive chemicals utilised in the grafting process and the catalyst masterbatch production.

1 GRAFT COPOLYMER

This is the most critical component of the whole process and can only be made on sophisticated extrusion compounding equipment that incorporates advanced process control systems. This stage of the process demands much higher levels of technology than normal compounding processes. The controls and technology are necessary because in this process the compounding equipment is being used as a chemical reactor in addition to its normal functions of mixing, dispersing and pumping. The higher level of technology has also been applied to product packaging to guarantee long term storage and protection of the product.

2 CATALYST MASTERBATCH

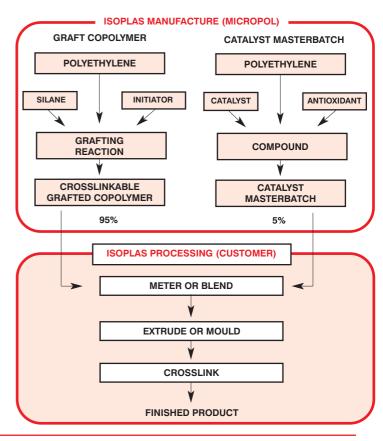
The range of ISOPLAS catalyst masterbatches produced by Micropol is manufactured on simular high technology compounding equipment. This ensures that the **catalyst** itself is consistently dosed and dispersed. The function of the **catalyst** is to accelerate the crosslinking of the **graft copolyme**r by the chemical action of water in the water bath or steam chamber.

In addition to the catalyst this masterbatch may contain process and product stabilisers to protect the pipe at the extrusion stage and in service. **Process aid** can be incorporated to improve the flow, extrusion quality and gloss of pipes produced, and even coloured pigments can be added, matched to customers' requirements. Micropol recognise that ISOPLAS will be processed on a range of equipment with different capabilities and mixing options, so we have developed a wide range of catalyst masterbatches to give the best possible results during production.

GUIDE TO PRODUCTION AND PROCESSING

PROCESS EQUIPMENT

Fabrication of crosslinkable components such as pipes, injection mouldings etc. is carried out on a wide range of conventional equipment. In contrast to other types of crosslinkable polyethylene, **no special equipment modifications are required**. ISOPLAS will process similarly to conventional polyethylenes of comparable melt flow index and density. A blend of 95 parts of graft copolymer and 5 parts of catalyst masterbatch (together with any colour masterbatch required) is prepared by either tumble blending or metering and this is simply extruded, injection moulded, extrusion blow moulded etc. A guide to typical process conditions and equipment follows:-



PRODUCTION ADVANTAGES

ISOPLAS can simplify, reduce costs and improve safety standards of production in comparison to peroxide crosslinked polyethylene in several ways:-

- 1) No health hazards are present at any stage of processing, crosslinking or end usage.
- 2) Separation of the fabricating and crosslinking steps avoids the use of complicated continuous vulcanisation systems which in turn will yield the following advantages.
- 3) Output rates can be selected independent of the crosslinking rate and so maximum production rates can be achieved.
- 4) Temperature profiles can be set without the fear of premature crosslinking occurring somewhere in the processing equipment.
- 5) Start-up scrap and out of tolerance material is kept to a minimum.
- 6) Maintenance downtime is reduced due to the simpler nature of the equipment.

	EQUIPMENT	PROCESS TEMPERATURES	PRODUCTION RATES
PIPE EXTRUSION	Conventional single screw extruders with single or twin start polyethylene screws. L/D ratios preferably 25:1 minimum for good mixing characteristics. Obstructions to straightforward melt flow paths, such as mixing tips, basket dies, restrictor blocks, etc. should be avoided.	Barrel 150-180°C Head/Die 190-210°C Hopper throat cooling desirable	For 10-30m/min pipes typical line speeds are from 10-20 m/min on outputs from 50-200 kph
INJECTION MOULDING	Screw preplasticising machines capable of running low melt flow materials. Generously proportioned runners and gates. Hot runner systems can be used.	Plastication temperatures of 175-225°C on the barrel.	Comparable with moulding conventional HDPE injection grades.

CROSSLINKING

Crosslinking is carried out off line after processing by a simple batch process of exposure to water at elevated temperatures. Diffusion of trace amounts of water into the article, together with the action of heat, acts via hydrolysis and condensation of the alkoxy groups to form siloxane crosslinks. The presence of the catalyst from the masterbatch accelerates this process. Crosslinking can be achieved by:-

- 1) Immersion in hot water;
- 2) The action of low pressure steam;
- 3) Circulation of hot water through the interior of pipes.

The time to complete crosslinking is reduced by:
1) Increase in temperature of water/steam;

- 2) Reduction in thickness of section to be crosslinked;
- 3) Reduction in density of Isoplas grade;
- 4) Modification of the catalyst system supplied.

The rate of crosslinking depends on the rate of diffusion of water molecules into the wall of the article. This in turn is proportional to the square root of the wall thickness. In practice, if the wall thickness is doubled then the time for full crosslinking is increased fourfold. Crosslinking by exposing both sides of the article's surface to water/steam is therefore much more efficient than from one side only. Rate of crosslinking graphs for the major ISOPLAS grades at a range of water temperatures are available from Micropol's technical department.

HOUSE KEEPING

1) Storage

The two components of ISOPLAS should be stored separately in cool, dry conditions to prevent crosslinking before processing. After fabrication no special storage precautions are necessary.

2) Rework

In common with other crosslinkable polyethylenes ISOPLAS scrap material is not re-useable or recyclable.

3) Purging and Cleaning

After short production stops, run ISOPLAS to waste for a short period. After longer stops and after all production runs the extruder should be purged with stiff flow conventional HDPE. Die head, screw and barrel should then be stripped down and physically cleaned.

PACKAGING

ISOPLAS P is supplied in the form of two granular components. Graft copolymer is packed inside vacuum-sealed aluminium-laminated sacks inside 500 Kgs or 1 tonne octabins. This product is therefore protected against moisture ingress which could trigger premature crosslinking of the granules.

Catalyst masterbatch is packed in 25Kgs HDPE sacks. This product is not chemically affected by moisture vapour.

PROPERTIES

Fully crosslinked finished articles made from ISOPLAS crosslinkable polyethylene will exhibit outstanding resistance to rigorous environments. The major areas of property improvement in comparison with conventional and many other thermoplastics are:-

IMPROVED HEAT RESISTANCE
IMPROVED OXIDATION RESISTANCE
IMPROVED WEATHERING RESISTANCE
IMPROVED LOW TEMPERATURE STRENGTH
IMPROVED CHEMICAL RESISTANCE
IMPROVED ENVIRONMENTAL STRESS CRACK RESISTANCE
IMPROVED LONG TERM STRENGTH AT ELEVATED TEMPERATURES
IMPROVED STRESS RESISTANCE AT ELEVATED TEMPERATURES
IMPROVED PERMEATION RESISTANCE

The results of detailed studies into the property achievements described above are available from Micropol's Technical Services Department.

One example of the improved performance of ISOPLAS GRADES is given by data on STRESS RUPTURE AT ELEVATED TEMPERATURES.

The most important property of a pipe is its pressure performance. This property is an outstanding feature of ISOPLAS crosslinkable polyethylene's performance and has been a major reason for its acceptance in the pipe industry in Europe for underfloor heating and domestic hot and cold water pipe systems. The improvement in comparison with conventional polyethylene has allowed it to be used under conditions previously thought to be beyond any polyethylene material.

Long term stress rupture tests on ISOPLAS at 95°C have so far failed to reproduce conventional polyethylene's characteristic fall or "knee". Thus the life of ISOPLAS crosslinkable polyethylene pipes can be successfully extrapolated beyond a design lifetime of 50 years.

Compared with other thermoplastic materials that could be used for pipes to carry hot water, ISOPLAS crosslinkable polyethylene has the major characteristic of the best predictable long term stress rupture resistance. In addition the change from a thermoplastic to a thermoelastic structure considerably improves its performance over polypropylene and polybutylene which will always retain their thermoplastic behaviour. Together with this exceptional stress rupture resistance other improved properties such as resistance to oxidation and outstanding thermal stability have led to ISOPLAS finding an increasing number of outlets in high quality systems and installations where long term security is essential.

Our current customers are offering 10 year warranties on pipe made from ISOPLAS for use in underfloor heating and are guaranteeing a permitted temperature range of -60°C to +92°C under a continuous pressure of 3 bar and are allowing for 50 years working life at conditions within this range. They are also guaranteeing short term exposure to 110°C at the same pressure. Pipe systems based on ISOPLAS ensure optimum quality and safety.

ISOPLAS Grades designed for central heating systems and for hot sanitary water applications have been tested in pipe form at 110°C and have not failed at pressure exceeding the requirements of DIN 16892 at periods well over double the 8000 hours requirement of this standard. Regression curves for individual ISOPLAS grades are available from Micropol.

POTABLE WATER CONTACT APPROVALS HELD BY MICROPOL

Water Research Council UK, Water Regulations Advisory Scheme Water Fittings and Materials Directory Reference Numbers.

P471 0310502 P501 0308513 P602 9909519

Customers of Micropol have their own approvals from the following countries:

Italy Gazette Ufficiale No. 104

Certificate of the Laboratorio di Igiene e Profilassi di Milano

France L'Institut D'Analyses et D'Essais du Centre-Ouest

Germany D.V.G.W. K.T.W. Certificate

Switzerland Bundesamt für Gesundheitswessen (B.A.G.)

Hungary Staat Liches fur Umweltschultz Portugal Instituto Nacional de Saude Belgium Examen des Polluant de l'Eau

USA NSF International ANSI/NSF Standard 61

Australia AS 3855 or AS 4020

Formulations now available to meet the new

EAS (harmonised) water regulations.

Details of the approvals and the various migration limits imposed by the National authorities are available.

PROPERTIES

Physical Properties	Test Method	Units			Grade Ra	ange	
Mechanical			P381	P471	P501	P651	P602
Density	ASTM D792	g/cm³	0.944	0.947	0.952	0.960	0.964
Tensile Strength at Yield	ASTM D638	MN/m ²					
at 23°C			23.0	24.0	26.0	31.0	31.0
at 100°C					6.5	9.0	
Elongation at break	ASTM D638	%	250	200	200	70	250
Modulus of Elasticity	ASTM D638	MN/m²					
at -40°C					1400	2000	
at 0°C					1200	1500	
at +20°C			1100	780	1000	1800	1750
Thermal							
Melt Flow Index	ASTM D1238 190/2.16	g/10 mins					2.0
	190/5		1.75	0.30	5.0	1.5	8.0
Vicat Softening Point	ASTM D1525	°C	126	120	124	130	127
Specific Heat		KJ/kg/°C	1.9	2.0	2.1	2.1	2.0
Coefficient of Linear Expansion	ASTM D696						
at -20°C		PER °C			9.0x10 ⁻⁵	9.0x10 ⁻⁵	
at +20°C				1.3x10 ⁻⁴	1.4x10 ⁻⁴	1.4x10 ⁻⁴	2.5x10 ⁻⁴
at 100°C					5.0x10 ⁻⁴	5.0x10 ⁻⁴	
Thermal Conductivity	BS 874	cal/s/cm/°C	1.03x10 ⁻³	1.03x10 ⁻³	1.1x10 ⁻³	1.1x10 ⁻³	1.03x10 ⁻³

APPLICATION AREA:

P602 - Injection mouldings

P381 - Pipes for underfloor heating P501 - Pipes for Hot Sanitary Water P471 - Alupex Pipes

P651 - Rigid pipes for District Heating Systems

The information given above is typical for the material. It should only be used to compare one material with another and does not guarantee performance under end-use conditions.







Bayley Street, Stalybridge, Cheshire, SK15 1QQ Tel: 0161-330 5570 Fax: 0161-343 7687

email: microlink@micropol.co.uk Website: www.micropol.co.uk



ISOPLAS INFORMATION GUIDES

INDEX

Guide	Title
1	THE EXTRUSION OF PIPES AND TUBES
2	HOUSEKEEPING PROCEDURES
3	INJECTION MOULDING
4	STANDARD PROPERTY TESTING PROCEDURES
5	CROSSLINKING ISOPLAS
6	PROCESS AID
7	LONG TERM PRESSURE TESTING
8	CREEP DATA
9	EXTRUSION EQUIPMENT SUPPLIERS - CONVENTIONAL (NON-COMPOSITE) PIPES
10	EXTRUSION EQUIPMENT SUPPLIERS - CO-EXTRUDER DIE HEADS
11	EXTRUSION EQUIPMENT SUPPLIERS - ALUMINIUM CO-EXTRUSION LINES
12	MATERIAL SUPPLIERS - POLYMERIC OXYGEN BARRIER & ADHESIVE TIE LAYERS
13	APPROVAL FOR DRINKING WATER CONTACT – MAJOR SPECIFYING AUTHORITIES & TEST CENTRES; EXTRACTION FOR APPROVAL
14	PIPE AND TUBE EXTRUSION - DIE AND CALIBRATOR SIZES, DIMENSIONAL CONTROL
15	CHEMICAL RESISTANCE & ENVIRONMENTAL STRESS CRACK RESISTANCE

THE EXTRUSION OF PIPES AND TUBES

1. As far as operating the pipe extrusion process is concerned, there is no difference between the use of Isoplas 'P' and conventional polyethylenes of similar melt index and density.

After extrusion has been completed then the coils of pipe or tube lengths are placed in tanks of hot water **or** in a low-pressure steam sauna **or** steam/water may be circulated through them. The action of the water chemically crosslinks the pipes. The crosslinking can be carried out on batches of pipes at any time **after** the extrusion stage. During the extrusion process the extruder conditions may be adjusted in the normal way to achieve best pipe surface finish, calibration, throughput, etc., without affecting the subsequent crosslinking process.

- 2. The Isoplas material comes ready for processing. It is supplied in two parts:-
 - A. Isoplas P Graft copolymer (usually supplied in octabins). This is used at 95% in the extruder.
 - B. Isoplas Catalyst Masterbatch (supplied in clearly labelled 25kg plastic sacks).
 This is metered and blended with item (A) at 5% before extrusion.

At present there is a 3-month shelf life on the Isoplas graft co-polymer. The "use by" date is clearly marked on the outside of the octabins.

- 3. Micropol has four basic Isoplas 'P' grades for pipe according to application.
 - For under-floor heating pipes Isoplas P381
 - For composite aluminium/crosslinked polyethylene heating pipes Isoplas P471
 - For hot sanitary water pipes Isoplas P501
 - For rigid pipes for water transport and general-purpose use Isoplas P651.

4. Extrusion equipment

4.1. Extruder screws - Any screw designed for polyethylene extrusion is suitable for Isoplas P. No special design is necessary.

In practise, it is found that a minimum L/D ratio of 25:1 and a compression ratio between 2:1 and 3:1 gives the best quality extrusions.

- 4.2 Screw configuration As for a typical HDPE screw, ideally three zones feed, compression, metering. Feed zone and metering zone should be constant flight depth. Compression zone should taper from feed to metering over at least three turns. However, some of our customers have run Isoplas on constant taper screws, PVC screws and double helix (BK) screws. Basically, if the screw will process conventional HDPE satisfactorily then it will process Isoplas.
- 4.3 Mixing Tips, sometimes called "hedgehogs" or "pineapples", can cause hang-ups and should be avoided. A smooth screw path free of obstructions or dead spots is best.

4.4 Die Head Design

- a) A simple breaker plate with no screen pack is best.
- b) A simple torpedo die is preferred. Spiral dies have been used successfully. Basket dies (because of the more complicated/restricted flow path) are not usually successful.
- c) Land length: ten times (10 x) the pipe wall thickness seems about right for land length of die.
- d) Co-extrusion dies with complicated flow paths sometimes necessitate the use of **Micropol Process Aid** to aid flow through the lead. Information Guide No. 6 details the use of "P.A." in extrusions.
- To process and crosslink this material you will need the following additional equipment:
 - a) A gravimetric metering device into the hopper for the catalyst masterbatch. It is useful if this has two stations so that colour masterbatch can be added if required.

Or a simple tumble blender if a gravimetric metering device is not practicable. b) A hot water tank for crosslinking. This should be able to heat the water to at least 90°C and should hold several coils of pipe.

<u>Or</u> a source of hot water (around 90°C) for pumping through pipe coils.

<u>Or</u> a low-pressure steam sauna for carrying out the crosslinking.

<u>Or</u> a supply of steam for circulating through the pipe coils.

More detailed guidance on crosslinking is given in **Information Guide No. 5.**

- 6 Extrusion Processing Temperatures
 These will depend on the type of screw used and the die head configuration, but typically one would expect:
 - 6.1 Barrel Temperatures 150 (zone 1) to 190°C (last zone) with an even temperature gradient along the barrel.
 - 6.2 Throat Water Exit Temperature 20 30°C for consistent feeding.
 - 6.3 Die Head Temperature 190 220°C; as low as possible consistent with smooth extrudate with no melt fracture.
 - 6.4 Die Tip Temperature 220 240°C to give a smooth external and internal finish to the tube.

HOUSEKEEPING PROCEDURES

1. STORAGE

1a Graft copolymer grades – Isoplas P501/ P381/P471 etc.: keep in a cool dry area.

1b Catalyst masterbatch – Isoplas P511-CMB/P381-CMB etc.: keep in a cool dry area.

2. SHELF LIFE – GRAFT COPOLYMER

All graft copolymers have a shelf life of 3 months. The "use by" date is stamped on the large blue Isoplas labels attached to the sides of boxes or pallets.

Isoplas Graft /copolymer is normally supplied in 1000 kilo octabins. The granules are vacuum sealed inside a single aluminium laminated liner bag. This packaging prevents water vapour ingress, which could otherwise cause premature crosslinking in storage.

Unopened containers of graft copolymer can be utilised after the "use by" date has expired and good quality product will normally be obtained. Micropol however do not guarantee raw material properties after this date.

Once the packaging has been opened it is recommended that the graft copolymer be used within 24 hours. This same recommendation applies to blends of graft and catalyst masterbatch, e.g. blends left in hopper or intermediate containers.

3. SHELF LIFE CATALYST MASTERBATCH

The Isoplas catalyst masterbatches do not have any shelf life restrictions provided they are kept in dry condition.

In all cases it is recommended that correct stock rotation be applied, i.e. batches of graft and catalyst masterbatch are used in the order in which they are received by the customer.

The best practise for the highest quality extrusions is to order the quantity needed for each production run and to extrude it as soon as possible after arrival.

4 PREDRYING

There should be no need to pre-dry (e.g. by use of a hopper dryer) either graft or catalyst masterbatch. Both these materials are dried and tested to a very low level of moisture (water) content at Micropol's production facility.

Where extrusion takes place in locations of exceptionally high humidity levels, pre-drying of the graft / catalyst masterbatch has sometimes been claimed to improve extrusion quality. If this is undertaken, then the pellets should not be heated to greater than 70°C.

5 COLOUR MASTERBATCHES

Isoplas pipes can be coloured with good quality LDPE based pigment masterbatches. Micropol recommend pre-drying these masterbatches before use to remove any water content. This procedure is essential with carbon black masterbatches.

Suppliers of colour masterbatches are indicated under *Isoplas Information Guide 16.*

Colour masterbatches containing Titanium Dioxide (the majority do) may use Alumina or Silica coated TiO₂. There is some evidence that these coatings may release trace amounts of water during the melt extrusion process. For the best quality we recommend that "low alumina" or "alumina free" or "organic coated" pigment based masterbatches are purchased.

6. STOPPING AND STARTING

It is important for all extrusion operators/supervisors/management to understand that the combination of Isoplas graft copolymer and catalyst masterbatch in the melt phase inside an extruder is potentially a reactive mixture. If the extruder is stopped then the melt inside the barrel and die head will start to react with traces of water drawn in from the factory air through the feed hopper and will slowly begin to crosslink. This initially results in an increase in melt viscosity (it becomes stiffer) but after some hours full crosslinking will take place and the melt becomes solid and non meltable.

For short planned or unplanned stops of up to five (5) minutes duration it will be possible to restart with no special precautions other than **emptying** the extruder, although it is **always** best to keep extruders on

"trickle feed" if possible. For all planned **longer** stops, the extruder should be emptied and **purged out immediately**. The die head should then be separated from the barrel and cleaned out, and the barrel should be emptied.

For purging compound HDPE grades of density minimum 0.950 and of MFI \leq 1.0 at 190°C/2.16 kilo load should be used. Pipe extrusion grades of HDPE are generally satisfactory.

If the extruder and die head are of simple straight forward design, i.e., no mixing tips, complicated die channel arrangements etc., then extrusion can restart using Isoplas after this simple purging process.

However it is strongly recommended after all stops of longer than 1/2 hours (e.g. weekend stops) that, after purging, the screw should be removed and the die head be stripped and that all metal surfaces of screw, barrel and die interior be physically cleaned. All purging, however efficient, tends to leave small quantities of Isoplas in "dead spots" in the melt flow path. After cooling and reheating, these small deposits can harden and detach themselves during later manufacturing and thus could cause wear spots in later pipe production.

As indicated above, if the melt flow route is very complicated with many potential dead spots then stripping and cleaning may have to be carried out after even short stoppages.

Each factory will arrive at different solutions to the everyday cleaning/purging routines according to its production plant, type of equipment etc. No Isoplas customer should have any difficulties provided they are aware of the basic nature of the material and take these common sense precautions.

7. DIE DEPOSITS

During the course of normal pipe extrusion a small deposit may build up on the die plate inside and outside the emerging pipe. This is quite normal and is a result of the chemistry of the Isoplas process. The die deposit is normally tapped off between coils of pipe. If this deposit becomes very heavy then we can advise on possible changes to die geometry or the use of process aids to reduce it.

INJECTION MOULDING

Several grades of ISOPLAS crosslinkable polyethylene are suitable and have been used for the production of injection mouldings.

The two main grades recommended are:-

ISOPLAS P501 - M.F.I. 2.0 at 190° C / 5 kilo load ISOPLAS P602 - M.F.I. 8.0 at 190° C / 5 kilo load

Both these grades have been used for the injection moulding of pipe fittings. Isoplas P602 has been specifically developed for this and, because of its higher melt flow index, can be used in a wider range of applications.

MOULDING PROCEDURE

- 1. Prior to moulding, 5% of Isoplas P511/CMB (Catalyst Masterbatch) must be tumble blended with either of the above two grades. The blend is then fed to the machine the catalyst masterbatch can alternatively be metered to the machine via standard dosing equipment (Gravimetric).
- 2. Typical injection moulding conditions that have been used to produce injection moulded pipe fittings are as follows:-

MACHINE	HOT RUNNER (°C)	E		L. TEM C)	Р	NOZZLE (°C)	INJ. PRESSURE (bar)
		1	2	3	4		
STORK REED 250 TONNE	NO	200	210	230	230	220	35
ARBURG 320 D	YES 210-220	180	190	190	200	-	40

- 3. Essentially as far as injection moulding is concerned the ISOPLAS grades will behave exactly as HDPE of M.F.I. 2 or 8 at 190°C / 5 kilo load with the following exception:-
- 4. If it is required to keep the machine **STOPPED** for more than 5/10 minutes we advise that the screw and barrel be purged with a low melt flow index HDPE.

This is because any material left in the barrel will slowly begin to crosslink. At moulding temperatures the combination of graft copolymer/catalyst masterbatch together with a small amount of moisture drawn in through the hopper from the ambient air will slowly begin to react. This initially just leads to an increase in melt viscosity but if left for a period of some hours the Isoplas would eventually totally crosslink and become non-meltable.

After any moulding run is complete, the screw and barrel must be purged and then the screw removed for cleaning (together with the inner surface of the barrel). This is to remove any trace of Isoplas not cleared by purging. Any residue left in the machine would otherwise slowly harden and could contaminate subsequent production runs.

Moulded articles produced from Isoplas are crosslinked in the usual way by immersing in hot water or steam. The time required to achieve full crosslinking will depend on the temperature of the curing medium used and the wall thickness of the moulding. Further information is available in Information Guide 5 - "Crosslinking of Isoplas".

However, two factors should be considered:-

- a) The crosslinking process itself causes a shrinkage of between 0.5% and 1.0%. This value should be added on to the normal HDPE shrinkage due to cooling from the mould of 1% to 3 %. In practice we recommend that trial mouldings are produced on existing tools of similar dimensions, and the part dimensions are carefully checked after crosslinking, before new tooling is specified. Moulds which have produced Polybutylene fittings have been successfully used to make Isoplas fittings without modification. No insuperable problems of ovality have been found with the addition of the crosslinking step.
- b) If the injection moulding is to be welded to other parts to form a final assembly, then the welding operation should be carried out before crosslinking. Isoplas mouldings of up to 50% crosslinking degree have been successfully welded, but above this the weld strength falls off. As Isoplas will slowly crosslink in storage, assembly operations should be scheduled within a reasonable time of injection moulding.

Micropol's laboratory can offer assistance by measuring % crosslinking on customers' mouldings at any stage in the moulding, assembly or crosslinking process.

STANDARD PROPERTY TESTING PROCEDURES

1. Incoming Isoplas deliveries

What testing is carried out on the granules of Isoplas graft copolymer before extrusion or moulding is determined by customers' Quality Control Departments.

The most frequently applied test is Melt Flow Index. The test method Micropol use for this property is based on ASTM D1238. This is equivalent to ISO 1133 or BS 2782-720A or DIN 53735.

Melt flow index is normally measured at 190°C using a 2.16, 5 kilo or 21.6 kilo load. It is important to realise, however, that Isoplas does not behave identically to conventional HDPE in this test, in that successive cut-offs at fixed intervals tend to reduce in mass due to melt viscosity increase with time in the barrel of the m.f.i. viscometer.

Micropol uses a manually operated m.f.i. grader and measures the first one minute cut-off for reproducibility. Test methods specifying a number of cut-offs and/or semi-automatic testing machines may give different values.

2. Tests on extruded or moulded parts

2.1 Degree of crosslinking

The most important parameter which customers will need to measure is the degree of crosslinking (often called gel content). Most standards for Silane grafted polyethylene pipes specify a minimum value of 65% and it is obviously necessary to check if the crosslinking process (immersion of the finished part in hot water or steam) has proceeded long enough to achieve this value.

It is often helpful to check from time to time the degree of crosslinking directly after processing to verify that the minimum possible level of crosslinking has occurred during the extrusion or moulding process.

The degree of crosslinking is measured by a method described in DIN 16892 Section 4.4. A translation of this test method is as follows; -

4.4 DEGREE OF CROSSLINKING

To determine the degree of crosslinking one determines the portion of the test sample insoluble in boiling xylene.

- 4.4.1 <u>The Sample</u> From the end surface of a section of pipe, samples are taken in the form of shavings of 0.2 ± 0.02 mm thickness (most effectively with a lathe). The width of the shavings should correspond to the wall of the pipe; the length should total at least the circumference of the pipe.
- 4.4.2 The Method The shavings are weighed to the nearest 1 mg (mass M1) and placed in a container made of wiremesh or perforated tin-plate. The container with the shavings is placed in boiling and technically pure Xylene. 1% antioxidant (2.2-Methylene-bis-4-methyl-6-tert-butylphenol*) is added to the solvent. This is then refluxed for a period of 8 hours ± 5 minutes. The container with the residue is then taken out of the still boiling solvent, cooled to room temperature and dried. The samples immersed in the xylene are dried for three hours at a temperature of 140°C in an oven working by circulating hot air and with ventilation. After cooling off at room temperature the mass of the residue (mass M2) is weighed to the nearest 1 mg.
- 4.4.3 <u>Evaluation</u> The portion of the Mass insoluble in Xylene corresponds to the degree of crosslinking G in % and is calculated according to the following equation:-

 $G = \frac{M2}{M1} .100$

* PLASTANOX 2246 or LOWINOX 22M46 - obtainable from:-

Chemische Werke Lowi GmbH Teplitzer Strasse Postfach 1660 D-8264 Waldkraiburg Germany

Tel: (49) 8638 6080 Fax: (49) 8638 608200 The apparatus required is:-

- Electric heating mantle(s) suitable for round-bottomed flasks. a)
- 250ml round-bottomed flask(s). b)
- Condenser(s) to fit round-bottomed flask(s). c)
- d) Balance capable of weighing up to 150 gms with an accuracy of
- Oven (air circulating) capable of heating articles up to (say) e) 200°C.
- f) Xylene (technically pure).
- Antioxidant as described. g)

Notes

- 1. In practice we have found that the test results when the antioxidant specified is omitted do not change appreciably from when it is present.
- 2. Wire mesh containers can be conveniently fabricated from 30 mesh stainless steel extruder screen. Three or four mesh "pouches" can be placed in one flask, each cut with a different number of notches at the sealing flap to aid identification. Micropol Laboratory can provide samples of these pouches. By arrangement we can also train customers' laboratory staff on our own premises in this and other test methods.
- 3. Customers' own samples can be tested by Micropol to build up rate of crosslinking graphs etc. More details are contained in Information Sheet 5, "Crosslinking of Isoplas".

2.2 **Pressure Testing**

Many pipe standards specify minimum times to failure at elevated temperatures and pressures for either type testing or routine quality control requirements.

Apparatus for pressure testing at temperatures from 20 to 110°C can be purchased from:

I.P.T.

Institut für Prüftechnik GmbH & Co. KG Schulstrasse 3 D-86447 Todtenweis Germany

Hammelmaskin Fabrik Industrivej 15 8450 Hammel Denmark

Tel.: (49) 8237 9660 Tel.: (45) 86961770 Fax: (45) 8696 2475

Fax: (49) 8237 966480

and

Renato Brignole s.r.l., Via Montebello 45 20025 Legnano

Tel.: (39) 0331 547.120 Milan Fax: (39) 0331 544 213 Italy

Pressure tests can be carried out to national standards, or to acquire design or regression data, by

Pipeline Developments Limited

Magnetic House 51 Waterfront Quay

Salford Quays

Salford Fax: (46) 155 26 31 25 M5 2XW

Tel.: (44) 161 877 8800

Fax: (44) 161 877 8855

S.K.Z.

Süddeutsches Kunststoff-Zentrum Frankfurter Strasse 15-17 D-97082 Würtzburg

Germany

Tel.: (49) 931 / 41 04-0 Fax: (49) 931 / 41 04-177

Materials Performance Inc. 1501 FM 2818 Suite 208

College Station TX 77840

USA

Tel.: (1) 409.764.2019 Fax: (1) 409.764.4001 Studsvik Polymer AB, S-611 82, Nyköping Sweden

Tel.: (46) 155 22 14 76

PPI

Plastics Pipe Institute

1275 K Street N.W. Suite 400 Washington D.C. 20005 U.S.A.

Tel.: (1) 202 3715306 Fax: (1) 202 3711022

2.3 For help and advice on tests required by standards authorities around the world, or on other internally developed useful quality control or design tests, please contact our Technical Service Department.

CROSSLINKING OF ISOPLAS

1. **General Principles**

1.1 Isoplas Graft Copolymer as supplied by Micropol is a graft copolymer of Silane and Polyethylene. It is *crosslinkable* polyethylene at this stage, i.e. the side chains of Silane grafted on to the polyethylene backbone have not linked with each other to form *crosslinked* polyethylene.

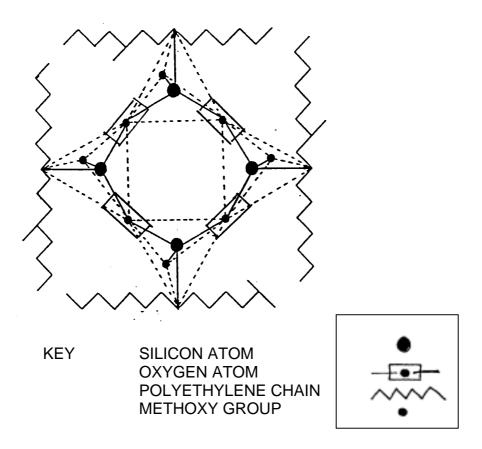
This graft copolymer is extruded or moulded, together with a catalyst masterbatch, under standard polyethylene processing conditions to form the finished part or article. At this stage (almost*) no crosslinking occurs.

1.2 Crosslinking takes place when the moulded or extruded article is exposed to water or water vapour. Molecules of water diffuse into the wall of the article and react with the Methoxy end groups on the Silane side chain first of all to form Hydroxy end groups:-

These hydroxy end groups either then react with other similar end groups to form a bridge or *crosslink* between the side chains and hence the polyethylene molecules:-

^{*} Because a small amount of water vapour is drawn into extruders/moulding machines via the hopper, a low level of crosslinking (up to 20%) does take place during the plastication process.

There are **three** end groups at the end of each Silane side chain; each can link with another side chain attached to another polyethylene molecule to form a three-dimensional polymer network:-



1.3 The function of the catalyst is to *increase* the *reaction rate* of the hydrolysis and crosslink-forming steps to ensure that full crosslinking takes place in as short a time as possible.

2 Crosslinking in practice

2.1 Before the crosslinks themselves can be formed water molecules have to diffuse into the wall of the article to reach the crosslinking sites. It is generally accepted that this diffusion process takes up most of the time necessary to achieve full crosslinking.

The higher the *temperature* of the water (or steam) in contact with the article to be crosslinked, then the higher the temperature the article itself will reach. As polyethylene increases in temperature, it expands and its density is reduced and the diffusion rate of water molecules into the plastic increases. The hydrolysis and crosslinking reaction rates will also increase (all chemical reactions speed up as temperature increases). So the time to full crosslinking will shorten.

At 20°C in air containing water vapour, a moulding of wall thickness 1.5mm reaches 60% crosslinking in 90 days. At 130°C in contact with steam under high pressure the time to 75% crosslinking is of the order of 90 minutes.

- 2.2 The thicker the wall of the article to be crosslinked, the longer water molecules will take to reach the centre and complete crosslinking. In practical terms, the rate of diffusion is proportional to the square of the wall thickness. So if wall thickness is doubled then it can take four times longer to complete crosslinking. By the same argument, crosslinking from both inner and outer surfaces of the article is faster than from one surface only.
- 2.3 After *crosslinking* is complete, the by-products of the crosslinking reaction should be *extracted* (by the same water molecules) from the wall of the tube or moulding. The main component to be extracted is Methanol.

If the article is to be used for drinking (potable) water applications, and therefore submitted for testing to National Water Authorities, the completion of the extraction process is important. Micropol can advise on the techniques and times needed to complete successful extraction as well as crosslinking to meet the different requirements of customers' National Water Authorities (see also *Information Sheet 12*)

2.4 Three main methods of crosslinking are in general use:-

2.4.1 Immersion in hot water

The articles are placed in stainless steel cages or baskets and lowered into stainless steel tanks containing water at as high a temperature as possible (80°C is the usual minimum, 95°C is better). The tanks are often sunk into factory floors for ease of handling and good insulation. Methanol extracted from the pipes is infinitely soluble in water, so normal top-up by floating ball valve to maintain water level is adequate. Pipe ends are kept open to allow water/hot moist air to penetrate the coils. After crosslinking, the pipe coils or mouldings are removed in their cages and allowed to drain in a well-ventilated area.

2.4.2 <u>Circulation of water and steam</u>

Hot water or steam under pressure is pumped through coils of pipe from a manifold, again at as high a temperature as practicable. For pipes containing an aluminium oxygen barrier this is the preferred method for crosslinking the inner layer. Sometimes steam can be allowed to vent into a chamber surrounding the articles to increase the rate of crosslinking. There is some evidence that circulating water is more efficient at extracting the reaction by-products than static water.

2.4.3 Steam sauna

Pipe coils or mouldings are placed on racks in a chamber filled with low pressure steam. Provided that the articles reach 100°C fairly quickly, this is quite an efficient method of crosslinking and extraction.

3.0 Typical crosslinking rates for Isoplas P501 pipes are shown in the table below:-

	WATER TEMPERATURE				
	80°C	95°C	95°C		
HOURS IN WATER	%XL	%XL	%XL		
0	21	17	27		
1/4	30	41	55		
1/2	32	50	61		
1	38	60	68		
2	47	69	68		
4	56	76	72		
8	65	76	74		
16	72	80	74		
24	74	81	75		
48	76	-	-		
PIPE WALL THICKNESS	3mm	3 mm	1.5 mm		

AIR TEMPER	RATURE
20°C	
%XL	DAYS IN AIR
19	2
23	4
32	6
38	10
45	20
50	30
54	40
56	50
58	60
60	90
1.5 mm	

Micropol Laboratory, by testing samples taken from customers' crosslinking plants at different times, can build up 'rate of crosslinking' graphs specifically for our customers' particular crosslinking processes.

4.0 Equipment suppliers

As yet, there appear to be no suppliers specialising in crosslinking tanks or installations. Sometimes capacity can be rented at local dye or chemical works, who may have stainless steel tanks or vats.

PROCESS AID - MICROPOL PA

1. APPLICATION

The function of Micropol PA is to lay down a thin layer of polyfluoroelastomer on the interior metal surfaces of the extruder. This layer, believed to be 50 micron thick, reduces the drag of Isoplas along these surfaces, which in turn reduces beard and gel formation.

Initially (for the first 15 - 30 minutes), a conditioning dose of 5% Micropol PA may be used. Once conditioning is complete the PA dose is reduced to 0.75 - 1.0%. In many cases our customers start up at this level and achieve the desired quality.

2. **GRADE REFERENCE:** Micropol PA

3. **PRODUCT TYPE**

Polyethylene containing a Fluoroelastomer designed for use at low addition levels to improve the processing of polyethylenes, particularly of high viscosity types.

4. CARRIER POLYMER

High density polyethylene of the following characteristic properties:

PHYSICAL PROPERTY	TEST METHOD	UNITS	VALUE
DENSITY	ASTM D792	g/cm ³	0.940
MELT FLOW INDEX	ASTM D1238	g/10 mins	8.000

5. **EFFECT ON PIPE OR MOULDING PROPERTIES**

We have not conducted specific trials on the effect of Micropol PA masterbatch on material strength. However, an increasing number of our customers are now using this process aid and (as is in the case of colour masterbatch) finding no adverse effect on regression curves, cyclic shock tests, long-term pressure testing, etc..

At 1% PA, the addition to the Isoplas melt is 0.02% active ingredient and 0.98% polyethylene carrier: at this level one would expect a negligible effect on final tube properties.

ISOPLAS P501 - LONG TERM PRESSURE TESTING

ISOPLAS is Micropol's grade specifically designed for the hot sanitary water market, i.e. for use in central heating systems and in hot potable water supply.

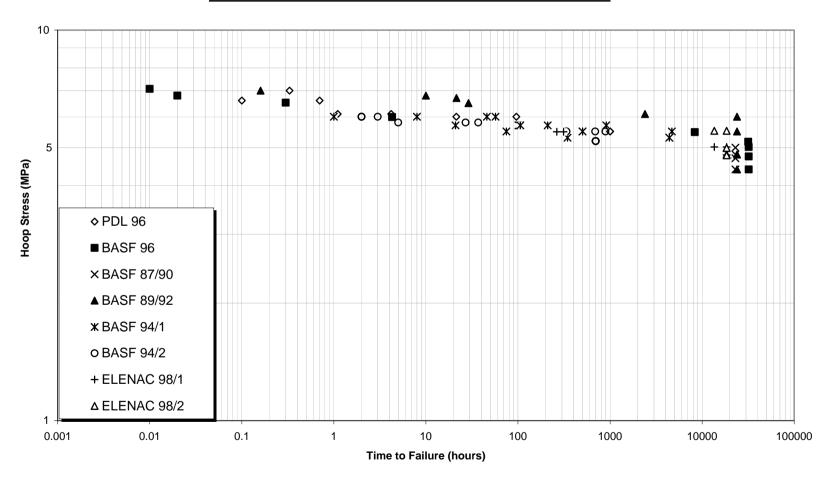
Because this is one of the more demanding applications, often involving customer guarantees to the installer and end users, an extensive programme of regression testing has been carried out at BASF and PDL (Pipe Line Developments Ltd) on fully crosslinked P501 pipes.

The results are summarised below in tabular and graph forms. One of the key requirements of DIN 16892 is for a pressure test at 2.8 MPa and 110°C to exceed 8000 hours without failure. It can be seen that in all three series of tests at this temperature Isoplas P501 exceeded this requirement comfortably.

HOOP STRESS REGRESSION DATA OF ISOPLAS P501 AT 95°C

a) PDL 96 b) 81%		a) BASF 96 b) 76%		a) BASF 89/92 b) 77%		
Hoop Stress (MPa)	Time to Failure (h)	Hoop Stress (MPa)	Time to Failure (h)	Hoop Stress (MPa)	Time to Failure (h)	
7	0.33	7.07	0.005	7	0.16	
6.6	0.1	6.8	0.02	6.8	10	
6.6	0.7	6.52	0.3	6.7	21.5	
6.1	1.1	5.99	4.3	6.5	29	
6.1	4.2	5.48	8328	6.1	2400	
6	21.4	5.18	>8760	6	>24000	
6	96.1	5.02	>9240	5.5	>24000	
5.5	1000	4.75	>9240	4.8	>24000	
5.5	1000	4.4	>9240	4.4	>24000	
a) BASF 94/1		a) BASF 94/2		a) BASF 87/9 b) 75%		
Hoop Stress		Hoop Stress		Hoop Stress		
(MPa)	Failure (h)	(MPa)	Failure (h)	(MPa)	Failure (h)	
6	1	6	2	5	>23000	
6	8	6	2	4.8	>23000	
6	8 46	6	3	4.8 4.7	>23000 >23000	
6 6 6	8 46 57	6 6 5.8	2 3 5	4.8	>23000	
6 6 6 5.7	8 46 57 21	6 6 5.8 5.8	2 3 5 27	4.8 4.7	>23000 >23000	
6 6 6 5.7 5.7	8 46 57 21 101	6 6 5.8 5.8	2 3 5 27 37	4.8 4.7	>23000 >23000	
6 6 6 5.7 5.7	8 46 57 21 101 103	6 6 5.8 5.8 5.8 5.5	2 3 5 27 37 337	4.8 4.7	>23000 >23000	
6 6 6 5.7 5.7 5.7	8 46 57 21 101 103 106	6 5.8 5.8 5.8 5.5 5.5	2 3 5 27 37 337 691	4.8 4.7	>23000 >23000	
6 6 6 5.7 5.7 5.7 5.7 5.7	8 46 57 21 101 103 106 210	6 6 5.8 5.8 5.8 5.5 5.5	2 3 5 27 37 337 691 893	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7	8 46 57 21 101 103 106 210 909	6 5.8 5.8 5.8 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.7	8 46 57 21 101 103 106 210 909 75	6 5.8 5.8 5.8 5.5 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700 700	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.7 5.5	8 46 57 21 101 103 106 210 909 75 502	6 5.8 5.8 5.8 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.5 5.5	8 46 57 21 101 103 106 210 909 75 502 504	6 5.8 5.8 5.8 5.5 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700 700	4.8 4.7	>23000 >23000	
6 6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.5 5.5 5.5	8 46 57 21 101 103 106 210 909 75 502 504 4700	6 5.8 5.8 5.8 5.5 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700 700	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.5 5.5 5.5 5.5	8 46 57 21 101 103 106 210 909 75 502 504 4700 345	6 5.8 5.8 5.8 5.5 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700 700	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.5 5.5 5.5 5.5	8 46 57 21 101 103 106 210 909 75 502 504 4700 345 4400	6 5.8 5.8 5.8 5.5 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700 700	4.8 4.7	>23000 >23000	
6 6 5.7 5.7 5.7 5.7 5.7 5.7 5.5 5.5 5.5 5.5	8 46 57 21 101 103 106 210 909 75 502 504 4700 345	6 5.8 5.8 5.8 5.5 5.5 5.5 5.5	2 3 5 27 37 337 691 893 700 700	4.8 4.7	>23000 >23000	

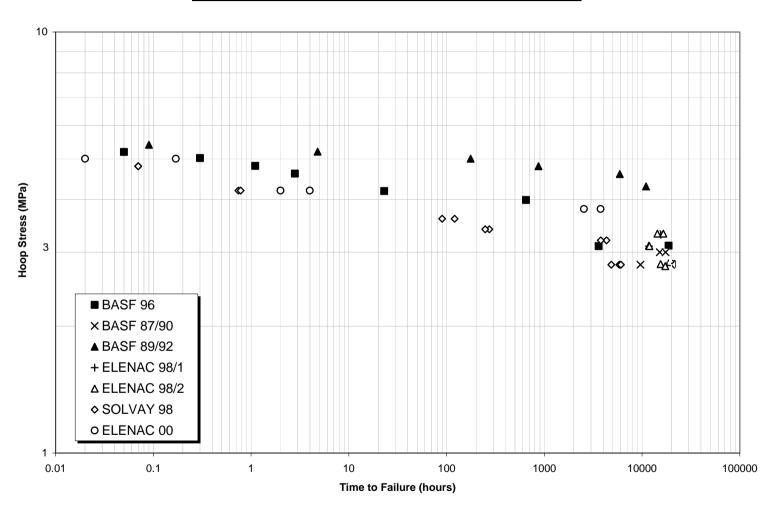
HOOP STRESS REGRESSION DATA OF ISOPLAS P501 AT 95°C



HOOP STRESS REGRESSION DATA OF ISOPLAS P501 AT 110°C

a) BASF 96 b) 76%		b) 77%		a) BASF 87/90 b) 75%			
Hoop Stress (MPa)	Time to Failure (h)	Hoop Stress (MPa)	Time to Failure (h)	Hoop Stress (MPa)	Time to Failure (h)		
5.19	0.05	5.4	0.09	3	15190		
5.02	0.3	5.2	4.8		17420		
4.81	1.1	5	176	2.8	19800		
4.61	2.8	4.8	870	2.8	9630		
4.19	23	4.6	5910				
3.99	650	4.3	10970				
3.1	3600	2.8	17860				
3.11	> 5292			_			
2.81	> 9240						

HOOP STRESS REGRESSION DATA OF ISOPLAS P501 AT 110°C



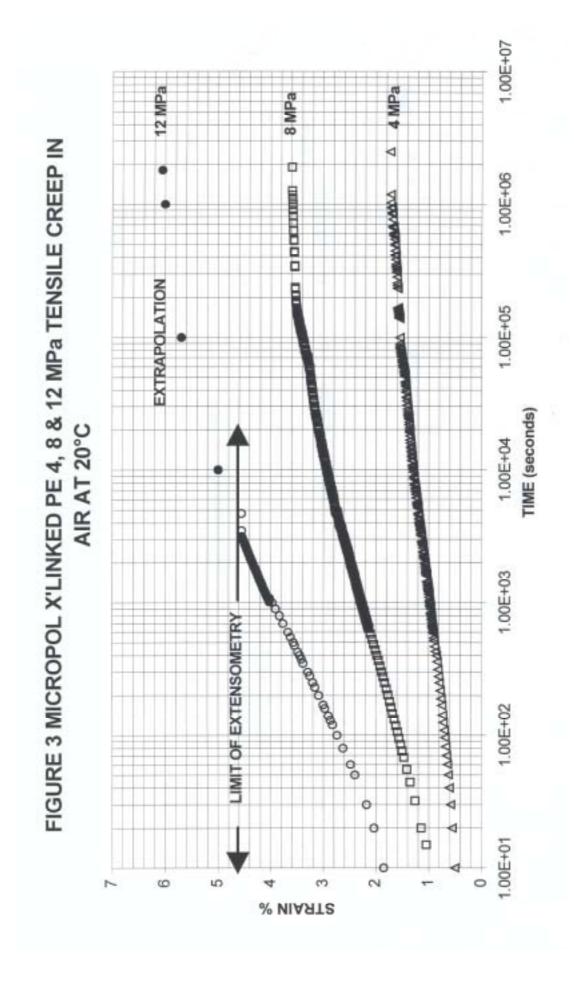
ISOPLAS P501 CREEP DATA

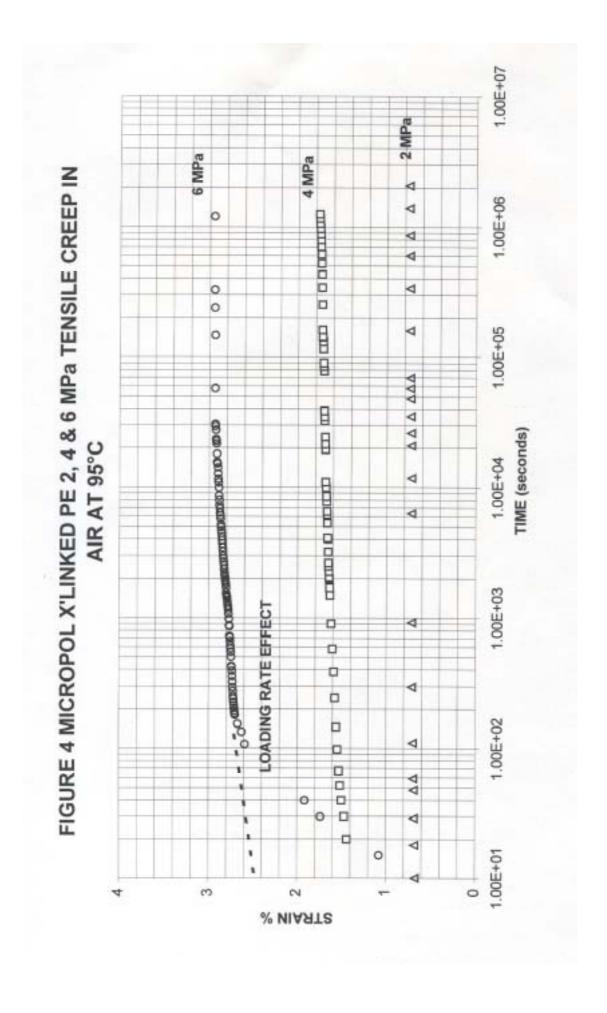
A programme of work was carried out at RAPRA Technology Ltd, on behalf of Micropol, on tensile creep on fully crosslinked Isoplas P501.

The samples tested were injection moulded ASTM D638 type IM tensile bars.

Tests were carried out at a range of stress values at 20°C and 95°C.

The creep test curves generated are published below. A full copy of the RAPRA test report is available on request.





ISOPLAS INFORMATION GUIDE 9

EXTRUSION EQUIPMENT SUPPLIERS CONVENTIONAL (NON-COMPOSITE) PIPES

From experience we have found the following equipment suppliers have supplied extrusion lines used satisfactorily with Isoplas Crosslinkable Polyethylene. This list is not intended to be exclusive and will be updated regularly in the light of our customers' experience.

Pipes produced have been either 100% Isoplas or, in conjunction with coextrusion dies and side arm extruders, multi-layer pipes containing polymeric oxygen barriers.

Amut s.pa. Via Cameri 16 28100 Novara

Italy

Tel: (39) 0321 6641 Fax: (39) 0321 474200

Battenfield Extrusionstechnik Extrusion Technology, Koningstraße 53,

D-32547 Bad Oeynhausen,

Germany

Tel: (49) 5731 2420 Fax: (49) 5731 27124

Hermann Berstorff Maschinenenbau

GmbH

An der Breiten Weise 305 PO Box 61 03 60

30635 Hannover

Germany

Tel: (49) 511 57020 Fax: (49) 511 5561916

Boston Matthews Machinery Ltd

Navigation Road

Diglis

Worcester, WR5 3DE

England

Tel: (44) 1905 763100 Fax: (44) 1905 763101

Cincinnati Milacron Austria GmbH

PO Box 111

Laxenburger Straße 246

A-1232 Vienna

Austria

Tel: (43) 1610 06112 Fax: (43) 1610 06292 Krause Maffei AG KM Straße 2 D-80997 München Germany

Tel: (49) 8988 990 Fax: (49) 8988 993092

Davis Standard Ltd 187 Stamford Way Sundon Way Sundon Park

Luton

Bedfordshire LU3 3AN Tel: (44) 1582 570501 Fax: (44) 1582 597363

Luigi Bandera SpA Corso Sempione 120 21052 Busto Arsizio,

Italy

Tel: (39) 0331 396111 Fax: (39) 0331 680206

Maillefer SA

1024 Ecublens-Lausanne

Switzerland

Tel: (41) 21 69 44 111 Fax: (41) 21 34 81 90

Reifenhäuser GmbH and Co.

Maschinenfabrik Spicher Straße 46-48 DE-53844Troisdorf

Germany

Tel: (49) 2241 4810 Fax: (49) 2241 40 87 78

See separate information guides for suppliers of multi-layer heads (Guide 10), polymeric oxygen barrier/adhesive materials (Guide 11) and aluminium/XLPE composite pipe lines (Guide 12).

EXTRUSION EQUIPMENT SUPPLIERS - CO-EXTRUSION DIE HEADS

Ridgeway Extrusion Technologists

Unit 22

W. and G. Estate

Challow Wantage

Oxon, UK Tel: +(44) 1235 760435 OX12 9TF Fax: +(44) 1235 763021

Microdia Tel: +(41) 24-447 35 00 Prés-du-Lac 69 bis Fax: +(41) 24-445 12 28

1400 Yverdon-les-Bains infos@microdia.ch

Switzerland

Nokia Maillefer SA 1024 Ecublens

Lausanne Tel: +(41) 21 69 44 111
Switzerland Fax: +(41) 21 69 12 143
mailleferextrusion.com

Bellaform

Ingelheim Tel: +(49) 6132 788 134 Germany Fax: +(49) 6132 788 285

bellaform.com

Reifenhaüser GmbH and Co. Maschinenfabrik

Spicher Straße 46-48

D-53844 Troisdorf Tel: +(49) 2241 4810 Germany Fax: +(49) 2241 408778 www.reifenhauser.com

This list is not comprehensive, but all the above have supplied co-extrusion die heads for the production of Isoplas pipes with polymeric oxygen barriers. Some companies will retro-fit such die heads to existing extrusion equipment.

EXTRUSION EQUIPMENT SUPPLIERS - ALUMINIUM CO-EXTRUSION LINES

All the companies listed below have supplied complete lines for the extrusion of composite tubes, consisting of an inner layer of Isoplas, and adhesive layer, an aluminium barrier layer, and adhesive layer and an outer layer of Isoplas or HDPE. Such tubes are also known as Alupex or Pexal.

Suppliers of aluminium strip for this process are also listed in this guide. The adhesives used are the same as those used in tubes with EVOH polymeric oxygen barriers and these are listed in *Information Guide 12*

1. COMPOSITE ALUMINIUM EXTRUSION LINES

Unicor GmbH Rahn Plastmaschinen

Industriestraße 56

D-97437 Hassfurt Tel: (49) 9521 690 122 Germany Fax: (49) 9521 690 195

This company also manufactures and sells Alupex pipes.

Ide Werkzeug - und Maschinenbau

Postfach 2105

D-7302 Ostfildern Tel: (49) 7158 1790 Germany Fax: (49) 7932 252

Kuhne Umtec GmbH

Postfach 3040

53739 St Augustin Tel: (49) 2241 311 081 Germany Fax: (49) 2241 902 180

Battenfeld Extrusiontech GmbH

Konigstraße 45 Postfach 100163

D32547 Bad Oeynhausen Tel: (49) 5731 2420 Germany Fax: (49) 5731 27124

Swiss Cab E. Kertscher S.A.

Rue de L'Industrie 5

PO Box 104

CH-1462 Yvonnand Tel (41) 2431 1502 Switzerland Fax (41) 2431 1447

2. Manufacturers of aluminium strip for composite AL-XLPE tubes

Granges Luxembourg Dudelange L3401

BP91 Tel (352) 518 664 Luxembourg Fax (352) 518 664 334

Reynolds Co Ltd Industrial Production Centre 5800 Keaton Crescent

Mississauga

Ontario Tel (1) 416 568 5325

Canada

MATERIALS SUPPLIERS -POLYMERIC OXYGEN BARRIERS & ADHESIVE TIE LAYERS

The most commonly used material for polymeric oxygen barrier layer is EVOH (ethylene-vinyl alcohol copolymer). This can be used (together with an adhesive tie layer) to coat the outside of an Isoplas tube. An alternative to this three-layer system is what is known as the "buried barrier" or 5-layer system. In this case the pipe structure is:

> inner layer -Isoplas

adhesive polymer tie layer -

barrier layer -tie layer -outer layer -**EVOH**

adhesive polymer

outer layer -Isoplas

The advantage of this system is that the oxygen barrier will not abrade or be damaged during installation or use. Typically, the EVOH oxygen barrier layer is 100 microns thick and the adhesive tie layer 50 micron. Sometimes the EVOH layer is coloured with masterbatch to show it is present and to increase opacity to light.

The three-layer system is simpler, with a lower capital cost for the die head. The outer layers can alternatively be applied by cross-head extrusion on to previously manufactured stock of Isoplas pipe.

For multi-layer materials we can offer the following information.

We have had most contacts with Elf Atochem, whose EVOH/adhesive system is running with Isoplas at several of our customers.

At Atofina, Centre d'Etude de Recherchement et Developpment, 27470 Serguigny, France, there is a demonstration 3/5 layer co-extrusion line on which you can see Atofina products Soarnol EVOH and Orevac Adhesive running with Isoplas. They offer materials/technical advice and practical knowledge of coextrusion, including running their demonstration extrusion line to purchasers of Atofina EVOH and adhesives.

Contact is:

Dr Denis Melot

Atofina

Centre d'Etude de Recherche et Développement (CERDATO)

27470 Serquigny

Eure Tel (33) 232 46 68 24 France (33) 232 46 69 51

E-mail: denis.melot@atofina.com

EVOH grades used have been Soarnol A4412

Soarnol DH4

Adhesive grades used have been **Orevac 18303S**

Orevac 18501N

Adhesive resins are also supplied by DSM.

Application expert is

Leo Kokelkoren (Technical Sales Manager)

DSM Engineering Plastics

Poststraat 1, Sittard Tel (31) 46 4762837 (Direct)

PO Box 43, 6130 AA Sittard Fax (31) 46 4770101

The Netherlands E-mail: leo.kokelkoren@dsm.com

Most-used grade is Yparex 8102.

Adhesive resins (ADMER) and EVOH (EVAL) are supplied by Mitsui and Kuraray, respectively.

Roman Jaroszewki (Manager, Technical Service, Specialty Polymers)

Mitsui Chemicals Europe GmbH

Oststrasse 10

40211 Düsseldorf Tel (49) 211 17 33 236 (Direct)

Germany Fax (49) 211 323486

E-mail jaroszewski@mcie.de

Mitsui Petrochemical Industries Ltd

Kasumigaseki Bldg.

2-5, Kasumigaseki 3-Chome

Chiyoda-ku

Tokyo 100 Tel (81) 580 3633 Japan Fax (81) 593 0813

Kuraray Co Ltd

Schiess Strasse 68

40549 Düsseldorf Tel (49) 211 538880 Germany Fax (49) 211 5388898

Kuraray Co Ltd 12-39, 1-Chome

Umeda Kita-ku

Osaka Tel (81) 348 2676 Japan Fax (81) 348 2563

Adhesive resins (Plexar) are supplied by Alphamin:

Jelmer Henskens (Speciality Waxes and Polymers) Alphamin Commercial and Development Centre

Avenue Pasteur 15

B-1300 Wavre Tel (32) 10 23 30 70 Belgium Fax (32) 10 23 30 71

E-mail: jelmer.henskens@alphamin.com

INFORMATION GUIDE 13

APPROVAL FOR DRINKING WATER CONTACT

MAJOR SPECIFYING AUTHORITIES AND TEST CENTRES

EXTRACTION FOR APPROVALS

General

In most countries where plastic pipes or tubes are used to transport hot or cold water for drinking purposes, standards exist to regulate the use of polymeric raw materials for these applications.

Such standards may be issued by the national standards body or the National Water Authority. Almost universally it is the Water Authority that has originally laid down the standard, and it is test houses designated by the Water Authority who carry out the test regimes on plastic pipes and fittings to determine whether they comply with the appropriate standard.

There is a wide divergence of standards and test methods used to assess whether a plastic article is suitable for drinking water contact. Most authorities specify a "conditioning" or "circulating and rinsing" regime as part of the test, to simulate the initial installation and testing of the articles when they are first installed in, say, a domestic supply in a house. After the appropriate number of extractions have been made the final water extract is then tested.

Most countries (with the exception of the USA) carry out a taste and smell test (usually with a panel of testers) to assess the organoleptic effect of the plastic part on the testwater.

Other tests on the final extract water sample can include:

growth of micro-organisms (UK) heavy metals contact (UK)

total organic carbon level (Germany)free chlorine level (Germany)

 G.C.-Mass Spectroscopy to identify all contaminants in a sample (USA)

Micropol has many years of experience in dealing with National Water Authorities and their designated test houses. This includes supplying (in confidence) to the test houses the full formulation details of all of our Isoplas grades.

We can offer help and advice on the procedures for submitting sample Isoplas tubes and fittings, etc., for drinking water testing in many countries.

Below are listed some of the approvals Isoplas grades have gained in various countries, a list of the major specifying authorities and test houses and copies of Micropol's own UK Drinking Water approvals.

1. Water Contact approvals held by Micropol – Isoplas.

Water Research Council UK, Water Byelaws Scheme. Entry No. 980 1502 (certificate attached).

2. Customers of Micropol have their own approvals from the following countries:

Italy Gazette Ufficiale No. 104

Certificate of the Laboratorio di Igiene e Profilassi di Milano

France L'Institut D'Analyses et D'Essais du Centre-Ouest

Germany D.V.G.W. K.T.W. Certificate

Switzerland Bundesamt für Gesundheitswessen (B.A.G.)

Hungary Staat Liches fur Umweltschultz

Portugal Instituto Nacional de Saude

Belgium Examen des Polluant de l'Eau

U.S.A. NSF International ANSI/NSF Standard 61

Australia AS 3855 or AS 4020

2. Major specifying and testing authorities and test houses.

UK Water Byelaws Scheme

WRc Evaluation and Testing Centre Ltd

Fern Close

Pen-Y-Fan Industrial Estate

Oakdale

Gwent Tel: (44) 1495 248454 NP1 4EH Fax: (44) 1495 249234

Approved Test Houses

ITS Cranleigh (UK) Ltd

Manfield Park Cranleigh

Surrey Tel (44) 1483 268800 GU6 8PY Fax (44) 1483 267579

Law Laboratories Ltd

Shady Lane

Great Barr

Birmingham Tel (44) 990 903060 B44 9ET Fax (44) 121 3667003 The Water Quality Centre

Thames Water Spencer House Manor Farm Road

Reading

Berkshire Tel (44) 118 923 6214 RG2 0JN Fax (44) 118 923 6373

WRc plc Henley Road Medmenham Marlow

Bucks Tel (44) 1491 571531 SK7 2HD Fax (44) 1491 579094

Germany DVGW Deutsche Verein des Gas und Wasserfachs e.V.

Postfach 14 03 62

53058 Bonn Tel (49) 228 9188-5

Fax (49) 228 9188-990

(Josef-Wirmer-Straße 1-3, 53123 Bonn)

Approved Test Houses

Technologiezentrum Wasser (TZW)

Karlsruher Straße 84 Tel (49)721 931630 D-76139 Karlsruhe Fax (49) 721 33160

Hygiene-Institut des Ruhrgebietes

Rotthauser Straße 19 Tel (49) 209 92 42 0 45879 Gelsenkirchen Fax (49) 209 92 42 105

Bundesansalt für Materialforschung und -prüfung

Unter den Eichen 87

12203 Berlin Tel: ((49) 30 8104 3135

Fax: (49) 30 811 2029

USA National Sanitary Foundation (NSF)

NSF International 3475 Plymouth Road

Ann Arbor

MI 48105 Tel (1) 313 769 8010 USA Fax (1) 313 769 0109

Netherlands

Kiwa N.V. Certificatie en Keringen Sir Winston Churchill-laan 273

Postbus 70 Tel (31) 70 395 3535 2280 AB Rijswijk Fax (31) 70 395 34 20

3. Extraction

As stated in *Information Guide 5*, it is important that Isoplas pipes manufactured for drinking water approval are fully extracted as well as crosslinked. the extraction process removes the by-products of the crosslinking reaction (mainly methanol) and ensures that the minimum level of substances is present in any subsequent extract being tested for drinking water contact.

It is Micropol's policy that all Isoplas formulations contain only B.G.A. (German Food Authority) positive list registered additives. Extensive analytical work in the UK and USA has ensured that levels of additive breakdown products in extracted water are very low indeed and these are toxicologically safe.

WATER BYELAWS SCHEME

Our Ref: CR/JCS

21 January 1998

Micropol Ltd **Bayley Street** Stalybridge Cheshire SK15 1QQ

Dear Sir





WATER BYELAWS SCHEME

"ITEMS WHICH HAVE PASSED FULL TESTS OF EFFECT ON WATER QUALITY - BS 6920"

We refer to your application for the material(s) described below to be approved arising from the results of the tests of effect on water quality that have been carried out on the product(s) so described, it has been decided that there is no objection to its/their use provided the source, nature and manufacturing processes of the ingredients and products are not changed. (See notes overleaf).

POLYETHYLENE - COMPONENTS

Isoplas P501. Translucent white crosslinkable polyethylene tube. For cold water and hot water use up to 85°C. This product is translucent and therefore not suitable for use in fittings where it maybe exposed to light.

Audit Test Report: MA2059/D

9801502

Micropol Ltd

An entry, as above, will accordingly be included in the Water Fittings and Materials Directory, Part Two, under the section headed, "Materials which have passed full tests of effect on water quality".

Your attention is drawn to the statement overleaf. Manufacturers or applicants may only quote in their sales literature terms which are used in this letter, namely that the product as listed, having passed the tests of effect on water quality, is suitable for use in contact with potable water and that a reference to the product will be included in the Materials section, Part Two, of the Water Fittings and Materials Directory: this may be abbreviated to "Water Byelaws Scheme - Approved Product" or "WBS - Approved Product". Approval of this product does not signify the approval of its mechanical or physical properties for any use.

The Technical Committee of the Scheme reserves the right to review approval. This product automatically becomes due for audit reassessment in January 2003.

Chris Ramsey (Miss) Materials Administrator Water Byelaws Scheme

WRc EVALUATION & TESTING CENTRE LTD, FERN CLOSE, PEN-Y-FAN INDUSTRIAL ESTATE,

OAKDALE, GWENT, NP1 4EH, UK.
TEL: 01495 248454. FAX: 01495 249234.
WRc Evaluation & Testing Centre Ltd. Registered in England No. 2







Our Ref: CR/JCS

19 August 1998

Micropol Ltd Bayley Street Stalybridge Cheshire SK15 1QQ



Dear Sir

WATER BYELAWS SCHEME "ITEMS WHICH HAVE PASSED FULL TESTS OF EFFECT ON WATER QUALITY - BS 6920"

We refer to your application for the material(s) described below to be approved arising from the results of the tests of effect on water quality that have been carried out on the product(s) so described, it has been decided that there is no objection to its/their use provided the source, nature and manufacturing processes of the ingredients and products are not changed. (See notes overleath.

POLYETHYLENE - COMPONENTS

isopias P471. Crosslinkable translucent white PE pipe. For cold water and hot water use up to 85°C. This pipe is translucent and therefore may not comply to BS2782 Part II. Should not be used in fittings where it maybe exposed to light.

Audit Test Report: MA2095/N

9805521

Micropol Ltd

An entry, as above, will accordingly be included in the Water Fittings and Materials Directory, Part Two, under the section headed, "Materials which have passed full tests of effect on water quality".

Your attention is drawn to the statement overleaf. Manufacturers or applicants may only quote in their sales literature terms which are used in this letter, namely that the product as listed, having passed the tests of effect on water quality, is suitable for use in contact with potable water and that a reference to the product will be included in the Materials section, Part Two, of the Water Fittings and Materials Directory; this may be abbreviated to "Water Byelaws Scheme - Approved Product" or "WBS - Approved Product". Approval of this product does not signify the approval of its mechanical or physical proportion for any uses or physical properties for any use.

The Technical Committee of the Scheme reserves the right to review approval. This product automatically becomes due for audit reassessment in May 2003.

Yours faithfully

Chris Ramsey (Miss) Materials Administrator Water Byelaws Scheme

WRC EVALUATION & TESTING CONTRE LTD, FERN CLOSE, PEN-YEAN INDUSTRIAL ESTATE, OAKDALK, GWENT, NP.1 4EH, UK. TEL: 01405 248454. FAX: 01495 249234.

Wife Bontonian & Treeton Course led, Repute







ISOPLAS INFORMATION GUIDE 14

PIPE AND TUBE EXTRUSION – DIE AND CALIBRATOR SIZES, DIMENSIONAL CONTROL

The following table shows the dimensions used on our laboratory pipe extruder. This may serve as a guide to process engineers. With different output rates, processing temperatures and extruders these may very well vary.

The two points to bear in mind are:

- 1. The draw down and post calibrator shrinkage due to cooling in the water tank are broadly similar to polyethylene of the same density as the Isoplas grade being used.
- 2. During crosslinking the pipe will shrink an additional 1% on length and ½ to 1% on diameter.

PIPE	FROM	AFTER	CALIBRATOR	PIN	DIE	SHRINKAGE
SIZE	EXTRUDER	XL	I.D.	O.D.	I.D	AFTER XL
(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(%)
7			7.8	8.1	12.3	
10	9.94	9.93	10.2	11.5	14.0	0.1
12						
15			15	18.5	24.9	
16			16.8	18.5	24.9	
18			18.9	20.4	27.9	
20			20.9	25.1	30.9	
22	22.26	22.04	22.8	25.7	31.9	0.9
28	28.40	28.04	28.9	28.5	34.9	1.2
30			30.4	38.0	45.9	
35						

Notes:

One suggestion may be to have the Pin O.D. on the large side and the die I.D on the small side so they can be tuned on production by removing metal.

The calibrators we use are made up of a series of spaced, concentric brass washers.

All the sizes quoted above have been derived empirically.

Land lengths for the die of approximately 10 x the wall thickness of the pipe to be processed have empirically given good results in terms of wall thickness consistency and low beard formation.

INFORMATION GUIDE 15

ISOPLAS - CHEMICAL RESISTANCE/ EVIRONMENTAL STRESS CRACK RESISTANCE

1. General

The silane crosslinking process, by increasing the molecular weight and the density of intramolecular bonds, gives some improvement on resistance and permeability to aggressive chemicals. A guide to chemical resistance of a typical uncrosslinked HDPE is included (Table 3) which can therefore be read as a minimum resistance table.

2.1 Chemical resistance – Petrol

Published papers on the subject of petrol resistance of silane-grafted polyethylene give the following information:

Table 1. Chemical resistance	Source: Polyethylene 1933 -1983)
------------------------------	----------------------------------

	Petroleum spir	it tests at 20°C	
Test period	Tensile strength	Elongation at	Weight change
	at yield	break	
DAYS	MN/m ²	%	%
0	30.5	250	-
7	24.9	425	+6.35
28	24.7	270	+6.39
63	25.2	360	+6.44
137	24.8	300	+6.58
185	25.8	240	+6.45

A small increase was observed in the weight of the samples due to absorption, but no significant changes were observed in the mechanical strength of the crosslinked polyethylene.

Permeation Resistance (Source: Dow Corning)

Testing with supergrade petrol at normal temperature carried out over a 45 day period showed that permeation was 25% less with Isoplas containers than those made from the best obtainable HDPE. The containers used had a wall thickness of 1.3mm and a capacity of 10 litres.

2.2 Chemical resistance – Freon refrigerant gases/liquids

Chemically Isoplas is resistant to refrigerant gases and liquids. Alupex pipe systems should therefore be suitable for this application.

3. Environmental stress crack resistance

Isoplas silane-grafted polyethylene displays outstanding E.S.C.R. properties compared to polythene.

Tests at BP Chemicals on containers moulded from Isoplas in contact with pressurised detergent solutions and field trials of Isoplas tubes containing hot water / ethylene glycol mixtures have confirmed this attribute.

Table 2 gives the results of some E.S.C.R. tests carried out by Dow Corning.

Table 2 Environmental Stress Crack Resistance

Test method ASTM D16933 (Bell Telephone Test) Time to Failure (Test Duration 7 months)

TEST SOLUTION	ISOPLAS P651	HIGH DENSITY PE
PETROL	> 7 months	66 hours
CONC. NITRIC ACID	5 days	66 hours
CONC. SULPHURIC ACID	21 days	-
DETERGENT SOLUTION	20 days	40 hours
BENZENE	> 7 months	90 hours
ENGINE OIL	> 7 months	138 hours

Guide to chemical resistance

Key

- **S.** Satisfactory. This chemical is only absorbed to a low level and thus has little or no measurable effect on physical properties.
- L. Limited resistance. A higher level of absorption occurs resulting in definite loss of physical properties. The question of the suitability of high density polyethylene would have to be considered with respect to the particular environment.
- E. Environmental stress cracking may occur.
- **NS.** Not satisfactory. Chemical attack or a high level of absorption occurs. In both cases the loss of physical properties is such that high density polyethylene is not suitable where prolonged contact is likely.

Substance	Conc.%	20°C	60°C
General chemical p	products		
Acetaldehyde	100	S	L
Acetic acid	10	S/E	S/E
Acetic acid	100	S/E	L/E
Acetic anhydride	100	S	L
Acetone	100	S	S
Acetophenetidin		S	S
Acetophenone	100	S	S
Acetylene	All	S	S
Acrylic emulsions	All	S/E	S/E
Acrylonitrile	100	S	S
Adipic acid		S	S
Aliphatic alcohols	100	S	S
Allyl chloride	100	S	S
Allyl alcohol	96	S	S
•			

Substance	Conc.%	20°C	60°C
Allyl chloride	100	L	L
Aluminium acetate	All	S	S
Aluminium carbonate	All	S	S
Aluminium chloride		S	S
Aluminium fluoride		S	S
Aluminium hydroxide		S	S
Aluminium nitrate		S	S
Aluminium oxalate		S	S
Aluminium oxychloride		S	S
Aluminium sulphate		S	S
Alums	All	S	S
Amino acetic acid	All	S	S
Ammonia (gaseous)	100	S	S
Ammonium acetate	All	S	S
Ammonium bicarbonate	All	S	S
Ammonium carbonate		S	S
Ammonium chloride		S	S
Ammonium fluoride		S	S
Ammonium hydroxide		S	S
Ammonium metaphosphate		S	S
Ammonium nitrate		S	S
Ammonium oxalate		S	S
Ammonium persulphate		S	S
Ammonium phosphate		S	S
Ammonium sulphate		S	S
Ammonium sulphide		S	S
Ammonium thiocyanate		S	S
Amyl acetate	100	L	L

Substance	Conc.%	20°C	60°C	Substance	Conc.%	20°C	60°
Amyl alcohol	10	L	L	Chloroethanol	100	S	S
Amyl chloride	All	L	L	Chloroform	100	NS	NS
Aniline	100	L	L	Chloromethane	100	L	_
Antimony	100	S	S	Chlorosulphonic acid	100	NS	NS
Antimony trichloride	100	S	S	Chrome alum		S	S
Aqua regia	100	NS	NS	Chromic acid	Sat.	S/L	NS
Arsenic	100	S	S	Chromium salts	Sat.	S	S
Arsenic acid		S	S	Citric acid		S/E	S/E
Aryl sulphonic acid	100	S	S	Copper salts (aqueous)	Sat.	S	S
Benzaldehyde	100	S	L	Cresols	100	S	S
Benzaldehyde (AQ)	Sat.	S	_	Cresylic acid	100	L	
Benzene	100	L	NS	Cresylic acid	50	S	S
Benzene sulphonic acid	100	S/E	S/E	Cyclohexane	100	L	L
Benzoic acid	Sat.	S	S	Cyclohexanol	100	S/E	S/E
Benzophenone	100	S	S	Cyclohexanone	100	S	L
Benzoyl cloride	100	L	L	Decahydronaphthalene	100	L	NS
Bismuth carbonate		s	S	Dibutyl ether	100	L	NS
Boric acid		S	S	Dibutyl phthalate	100	L	L
Boron trifluoride	100	S		Dibutyl sebacate	100	S	_
Bromic acid	10	S	S	Dichloroacetic acid	100	S	L
Bromine (liquid)	100	NS	NS	Dichloroacetic acid	50	S	S
Bromine (vapour)	High	NS	NS	Dichloroacetic acid/met		S	S
Bromine water	Sat.	NS	NS	o-dichlorobenzene	nyi estei	L	NS
Bromochloromethane	100	NS	NS	p-dichlorobenzene		L	NS
Butadiene	100	NS	NS	Dichloroethane	100	L	L
Butanediol	100	S/E	110	Dichloroethylene	100	NS	NS
Butane (gaseous)	100	S	S	Diethyl ether	100	L	L
Butane (liquid)	100	L	_	Diethylene glycol	100	S/E	S/E
Butanol	100	S	S	Diglycolic acid	100	S/E	S/E
Butoxyl	100		J	Dihexyl phthalate	100	S ·	S
(methoxy butyl acetate)	100	S	L	• •			
Butyl acetate	100	L	NS	Diisobutyl ketone	100	S	NS
Butyl alcohol	100	S/E	S/E	Dimethyl carbinol	100	S	_
Butylene glycol	100	S	S	Dimethyl formamide	100	S	L
Butyric acid	100	S	L	Dimethyl sulphoxide	100	S	S
Carbon dioxide (dry)	100	S	S	Dinonyl adipate	100	S	_
Carbon dioxide (wet)	100	S	S	Dinonyl phthalate	100	S .	L
Carbon disulphide	100	L		Dioctyl adipate	100	S	_
Carbon monoxide	100	S	S	Dioctyl phthalate	100	S	L
Carbon tetrachloride	100	NS	NS	1,4-Dioxane	100	S	8 -
Caustic potash (soln.)	50	S	S	Diphenyl ether	100	L	L
Caustic potash (soln.)	10	S	S .	Disodium phosphate	100	S	S
Caustic soda	50	S	S	Epichlorhydrin	100	S	S
Caustic soda	10	S	S	Epoxy resins	100	S	S
Chloral hydrate	100	S	S	Ethanediol	100	S	S
Chlorine gas (moist)		L	NS	Ether	100	S/E	L
Chlorine liquid		NS	NS	Ethyl acetate	100	S	L
Chlorine water		L	NS	Ethyl acrylate	100	L	L
Chloroacetic acid	100	S	S	Ethyl alcohol	100	S/E	S/E
Chlorobenzene	100	L	NS	Ethylbenzene	100	L	NS

Substance	Conc.%	20°C	60°C	Substance	Conc.%	20°C	60°C
Ethyl butyrate	100	L	NS	Iron salts (aqueous)	Sat.	S	S
Ethyl chloride	100	L	NS	Isobutyl alcohol	100	S	
Ethylene chloride	100	L	NS	Isooctane	100	S	L
Ethylenediaminetetra- acetic acid	100	S	S	Isopropanol	100	S	S
Ethylene dichloride	100	L	NS	Isopropyl ether	100	L	NS
Ethylene glycol	100	S/E	S/E	Lactic acid	100	S/E	S/E
2-Ethylhexanol	100	S	_	Lead acetate	Sat.	S .	S
Fatty acids (> 6)		S/E	S/E	Lead tetraethyl		S	
Ferric chloride		S	S	Magnesium carbonate		S	S
Ferric nitrate		S	S	Magnesium chloride		S	S
Ferric sulphate		S	S	Magnesium hydroxide		S	S
Ferrous ammonium citrate chloride	Sat.	S	S	Magnesium nitrate		S	S
Ferrous sulphate		S	S	Magnesium sulphate		S	S
Fluoboric acid	100	S	S	Maleic acid	50	S	S
Fluoric acid	40	S	S	Manganese sulphate		S	S
Fluorine	100	NS	NS	Mercuric chloride		S	S
Fluorosilicic acid		S	S	Mercuric cyanide		S	S
Formaldehyde (aqueous)	40	S/E	S/E	Mercurous nitrate		S	S
Formalin		S	S	Mercury	100	S	S
Formic acid		S/E	S/E	Methoxybutanol	100	S	L
Furfural	100	L	NS	Methyl acrylate		NS	NS
Furfuryl alcohol	100	S	L	Methyl alcohol		S/E	S/E
Gallic acid		S/E	S/E	Methyl bromide		L	NS
Glycolic acid	30	S/E	S/E	Methyl cyclohexane		L	NS
Glycolic acid butyl ester	100	S	S	Methylene chloride		NS	NS
Heptane	100	S	NS	Methyl ethyl ketone	100	S	L
Hexachlorobenzene	100	S	S	Methyl glycol		S	S
Hexamine	100	S/E	S/E	4-Methyl-2-pentanol		S	L
Hexane	100	NS	NS	Methyl sulphuric acid		S/E	S/E
Hydrazine	100	S/E	S	Monochloroacetic acid		S	S
Hydrazine hydrate	100	S/E	S	Monochlorobenzene		S	S
Hydrobromic acid	50	S	S	Naphthalene	100	S	
Hydrochloric acid	Conc.	S	S	Natural gas		S	S
Hydrocyanic acid	100	S	S	Nickel salts (aqueous)	Sat.	S	S
Hydrocyanic acid	Sat.	S	S	Nicotine	Dil.	S/E	S/E
Hydrocyanic acid	10	S	S	Nicotinic acid		S/E	S/E
Hydrofluoric acid	40	S	S	Nitric acid	50	L	NS
Hydrofluoric acid	70	S	S	Nitric acid	98	NS	NS
Hydrogen	100	S	S	Nitrobenzene	100	NS	NS
Hydrogen bromide	10	S	S	Nitroglycerine	100	NS	NS
Hydrogen peroxide	30	S	S	Nitropropane		L	L
Hydrogen peroxide	90	S	NS	o-nitrotoluene	100	S	L
Hydrogen phosphite	100	S	S	Nitrous gases	100	S	S
Hydrogen sulphide	Low	S	S	Nonyl alcohol	100	S	
Hydroquinone		S	S	Oleic acid	100	S/E	L/E
Hypochlorites	100	S	S	Oleum		NS	NS
Hypochlorous acid	All	S	S	Oxalic acid		S/E	S/E

Substance	Conc.%	20°C	60°C	Substance	Conc.%	20°C	60°C
Oxygen	100	S	S	Potassium perborate		S	S
Ozone		NS	NS	Potassium permangana	te	S	S
Palmitic acid		S/E	S/E	Potassium persulphate		S	S
Pentane		NS	_	Potassium sulphate		S	S
Perchloric acid	20	S	S	Potassium sulphide		S	S
Perchloric acid	50	S	S	Potassium sulphite		S	S
Perchloric acid	70	S	NS	Propane (gaseous)	100	S	S
Petroleum ether	100	S	L	Propane (liquid)	100	S	_
Phenol (aqueous phase)		S	S	Propargyl alcohol		S/E	S/E
Phenol (solid phase)	100	S	S	Propionic acid	50	S	S
Phenyl sulphonate		S	S	Propionic acid	100	S	L
Phosgene		S	_	Propyl alcohol	100	S/E	S/E
Phosphoric acid	25	S	S	Propylene dichloride	100	NS	NS
Phosphoric acid	50	S	S	Propylene glycol		S/E	S/E
Phosphoric acid	Conc.	S/L	NS	Pseudocumene		L	L
Phosphoric anhydride		S	S	Pyridine	100	S	NS
Phosphorous		S	S	Quinine		S	
Phosphorous oxychloride		S	٠L	Resorcinol	100	S	S
Phosphorous pentoxide		S	S	Salicylic acid		S	S
Phosphorous trichloride		S	L	Selenic acid	100	S	S
Phthalic acid	50	S	S	Silicic acid		S	S
Phthalic anhydride		S	S	Silicone fluids		S/E	S/E
Picric acid		S	S	Silver nitrate		S	S
Polyglycol ethers		S	S	Silver salts (aqueous)	Sat.	S	S
Potassium antimonate		S	S	Sodium acetate		S	S
Potassium bicarbonate		S	S	Sodium benzoate		S	S
Potassium bichromate		S	S	Sodium bicarbonate		S	S
Potassium bisulphate		S (*	S	Sodium bisulphate		S	S
Potassium borate		S	S	Sodium borate		S	S
Potassium bromate		S	S	Sodium bromide		S	S
Potassium bromide		S	S	Sodium carbonate		S	S
		S	S	Sodium chlorate		S	S
Potassium carbonate Potassium chlorate		S	S	Sodium chloride		S	S
		S	S	Sodium chlorite		S	S
Potassium chloride		_		Sodium cyanide		S	S
Potassium chromate		S	S	Sodium cyanide Sodium dichromate		S	S
Potassium cuprocyanide	:	S	S	Sodium dichromate Sodium dodecyl-		٠	J
Potassium cyanide		S	S	benzenesulphonate		S	S
Potassium dichromate		S	S	Sodium ferricyanide		S	S
Potassium ferri/ ferrocyanide		S	S	Sodium ferrocyanide		S	S
Potassium fluoride		S	S	Sodium fluoride		S	S
Potassium hydroxide		S	S	Sodium hydroxide		S	S
Potassium hypochlorite		S	S	Sodium hypochlorite		S	S
Potassium iodide	Sat.	S	S	Sodium hyposulphate		S	_
Potassium nitrate		S	S	Sodium nitrate		S	S
Potassium orthophosphat	te .	S	S	Sodium nitrite		S	S

Substance	Conc.%	20°C	60°C	Substance	Conc.%	20°C	60°C
Sodium orthophosphate	•	S	S	Xylene	100	L	NS
Sodium perborate		S	S	Zinc ammonium carbo	nate	S	S
Sodium peroxide	10	S	_	Zinc carbonate		S	S
Sodium peroxide	Sat.	L	_	Zinc chloride		S	S
Sodium phosphate		S	S	Zinc oxide		S	S
Sodium silicate		S	S	Zinc sulphate		S	S
Sodium sulphate		S	S				
Sodium sulphide		S	S	Oils and Waxes			
Sodium sulphite		S	S	Aniseed oil	100	L	NS
Sodium tetraborate		S	S	Beeswax	100	S	S
Sodium thiosulphate		S	S	Carnauba wax		S	S
Stannic chloride		S	S	Castor oil	100	S/E	S/E
Stannous chloride		S	S	Cedar leaf oil		NS	NS
Stearic acid	100	S/E	S/E	Cedar wood oil		NS	NS
Succinic acid	Sat.	S	S	Cinnamon oil		NS	NS
Sulphur		S	S	Citronella oil		NS	NS
Sulphur dioxide (dry)		S	S	Clove oil		S	S
Sulphur dioxide (wet)		S	S	Coconut oil		S	NS
Sulphur trioxide		NS	NS	Coconut oil alcohols		S/E	S/E
Sulphuric acid	10	S	S	Cod liver oil	100	S	_
Sulphuric acid	50	S	S	Corn seed oil		S	L
Sulphuric acid	95	S	L	Cotton seed oil		S/E	S/E
Sulphurous acid	30	S	S	Diesel oil		S	S
Sulphuryl chloride		NS		Fuel oil	100	S	L
Tannic acid		S/E	S/E	Kerosene		L	L
Tartaric acid	100	S	S	Lanolin	100	S	_
Tartaric acid (aqueous)	Sat.	S	S	Lemon peel oil		S	L
Tetrabromomethane	100	NS	NS	Linseed oil	100	S	S
Tetrachloroethane	100	NS	NS	Menthol	100	S	S
Tetrahydrofuran	100	L	NS	Mineral oil		L	L
Tetralin	100	S	L	Motor oil	100	S	L
Thioglycolic acid	100	S	S	Olive oil	100	S	S
Thionyl chloride		NS		Orange peel oil	100	S	_
Thiophene	100	L	L	Palm oil	100	S	_
Toluene	100	L	NS	Paraffin oil	100	S	L
Trichloroacetic acid	50	S	S	Paraffin wax	100	S	S .
Trichloroacetic acid	100	L	NS	Peanut oil	100	S	S
Trichloroethylene	100	L	NS	Peppermint oil		L	NS
Tri & chloroethylene				Petrol		S/L	S/L
phosphate	100	S	S	Pine needle oil	100	S	S
Tricresyl phosphate	100	L	L	Pine oil	100	L	NS
Triethanolamine		L	L	Salad oils	100	S	L
Urea		S/E	S/E	Silicone oil	100	S/E	L/E

Substance	Conc.%	20°C	60°C	Substance	Conc.%	6 20°C	60°
Soya bean oil	100	S	S	Pectin	Sat	S	S
Tallow	100	S	_	Pepper		S	S
Transformer oil	100	S	L	Pineapple juice		S	S
Turpentine oil	100	L	NS	Rum		S	S
White spirit		S/L	L	Saccharose	100	S	S
				Soda water		S	
Foods etc.				Starch		S	S
Beer		S	S	Sugar beet syrup	100	S	S
Brandy		S	_	Tea (leaves)		S	S
Butter		S		Tomato juice	100	S	S
Buttermilk		S		Tomato ketchup	100	S	S
Cider		S/E	S/E	Vanilla	100	S	S
Cinnamon		S	_	Vinegar	All	S	S
Cloves		S	_	Whisky	40	S/E	
Coca Cola		S	S	Wine (mulled claret)	100	S	S
Cocoa		S	_	Wine spirit	100	S	S
Coffee		S	_	Yeast	100	S	S
Cream		S	_				
Curds		S					
Dextrose		S	S				
Fructose		S	S	Car, garden, housel	hold pro	ducts	
Fruit juices		S	S	Antifreeze		S	S
Gelatine		S	S	Bleach		L	L
Gin	40	S	L	Borax		S	S
Glucose		S	S	Brake fluid	100	S/E	S/E
Glycerine	100	S/E	S/E	Brine		S	S
Glycerine (aqueous)	High	S	S/E	Creosote	100	S	S
Glycerine (aqueous)	Low	S	S	Cresol (aqueous)		S	S
Grapefruit juice		S	S	Detergents		S/E	S/E
Honey		S	S	Dextron		S	S
Horse radish		S	_	Emulsions			
Jam	100	S	S	(photographic)	100	S/E	S/E
Jelly	100	S	S	Floor wax	100	S	L
Lard		S		Furniture polish	100	S/E	L
Lemon juice		S	S	Hair shampoo		S	S
Lime juice		S	S	Hydraulic fluid	100	S	L
Margarine	100	S	S	Ink	100	S	S
Mayonnaise	100	S	_	Methylated spirit		S/E	S/E
Milk		S	S	Nail polish and remov	er 100	S	L
Molasses		S	S	Petroleum jelly	100	S	S
Mustard		S	S	Shoe polish	100	S	L
Orange juice		S	S	Talc		S	S
Paprika	100	S	S	Tar	100	S	L