Report on

CHLORIDE ION PENETRATION

INTO

CONCRETE CONTAINING

XYPEX

Report No : MR431

Date : March 1996

Client : Xypex Australia

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1. BRIEF

To carry out a test program on Xypex treated concrete to determine the effects on chloride ion diffusion characteristics that may result from the use of Xypex as an admixture.

To determine if early pore blocking in concrete will have any long term benefits in terms of durability.

To assess a number of other physical properties of concrete containing Xypex Admix, and to compare these with other concretes. These are -

- Compressive Strength
- Drying Shrinkage
- Flexural Strength

2. APPROACH

A series of comparative trials was deemed to be the best approach in determining the performance, and a number of concretes with known performance could be included in the trial series.

A series of three trials was ultimately chosen, as follows.

1.	Control mix -	Portland cement only, and no waterproofing additives
2.	Pore Blocking additive -	Portland cement only, with a pore blocking additive, based on ammonium stearate.
3.	Xypex -	Portland cement only with Xypex Admix

Samples were cast from these mixes, for testing to determine the resistance to chloride ion diffusion. This was done under a number of conditions, in an effort to model actual field conditions. To this end, diffusion samples were subjected to a series of cycles of immersion in salt water, followed by a period in air.

This approach is not normally taken in the assessment of chloride ion diffusion, as this characteristic is normally determined after a period of constant immersion. Whilst this provides a constant condition for the purposes of comparison, it does not indicate the type of performance may be achieved in, say, a tidal area. Various intervals were used in this testing, to ensure that performance with time could be determined.

To allow the relative performance of the Xypex treated concrete to be assessed against the recognised standard, a series of tests was carried out using constant immersion rather than cycling.

The most likely scenario in concrete that has to be protected in a marine environment is that it will receive an amount of curing (although usually not for a very long period), and then it will be exposed to the prevailing conditions. This will often mean a rising tide, when the concrete is still very young.

Durability is of increasing importance at present, and designers are looking for materials that will enhance durability performance. Accordingly, it has been suggested that pore blocking additives will be beneficial in enhancing the durability performance of concrete, because they will stop chloride ions entering (or moving through) the concrete at an early age (ie 2-3 days). Therefore the cycling process was, in come cases, commenced on stripping of the concrete, to model the likely conditions that the concrete would have faced had it been placed in a tidal zone.

A number of other properties of the various concretes would be determined, so that the possibility of the additives effecting performance in other areas could be determined.

3. TESTING

3.1 Trial Mixes

A series of three trial mixes was carried out in the laboratory, with the only variable being the durability additive that was used in the concrete.

The three mixes are shown in table 1, and it is clear from this that the binder contents, water-binder ratios and workabilities are all the same. Therefore relative performance of the additives can be assessed by making direct comparisons between the mixes.

Table 1 - Trial Mix Details

Mix	1	2	3
20mm (kg/m^3)	720	730	720
10mm (kg/m^3)	310	310	310
Coarse Sand (kg/m^3)	580	585	580
Fine Sand (kg/m^3)	195	200	195
Type SL Cement (kg/m^3)	365	370	365
Xypex (kg/m^3)	-	-	2.9
Pore Blocker (l/m^3)*	-	30	-
Superplasticiser (l/m^3)	1.0	1.2	1.4
Air Entrainer (ml/m^3)	100	100	100
Water/Binder	0.4	0.4	0.4
Density (kg/m^3)	2320	2330	2320
Air Content (%)	4.9	3.8	4.9
Slump (mm)	125	125	120

* This material is approx.70% water, and this water has been included in the free water used in calculating the water-binder ratio.

The mixes were made with commercially available materials, including Nepean aggregates and Type SL cement from a major supplier to the Sydney market.

3.2 Testing Regime

A series of samples was made from each mix, as set out in table 2. This also shows the test program.

Table 2 - Test Samples and Testing Regime

Samples	*Testing*
2-100mm dia. Cylinders	Compression testing at 7 days
2-100mm dia. Cylinders	Compression testing at 28 days
2-100mm dia. Cylinders	Compression testing at 56 days
1-100 x 350mm Beam	Flexural Strength at 28 days
1-100 x 350mm Beam	Flexural strength at 56 days
3-75mm Shrinkage Prisms	Drying Shrinkage to 56 days
5-150mmdia Cylinder	Chloride Penetration testing at various ages using various exposures

3.3 Chloride Penetration Testing

The testing for chloride ion penetration was carried out by immersing the samples into a bath of water maintained at a chloride ion content of 1.75%. The samples were each exposed to a range of cycles of immersion in and removal from the salt water bath, as described in table 3.

At the completion of the immersion cycles, the samples were face ground to remove increments (each of 2mm depth) of concrete dust. These dust samples were then tested for chloride ion content. This allowed the chloride ion profile of the samples to be plotted, and the diffusion rates of chloride ions into the concrete to be calculated under the various conditions.

Table 3 - Chloride Penetration Exposure Conditions

Sample	Exposure Condition
1	Samples cycled into and out of salt water (24hrs in &24 hrs out) for the 28 days immediately after casting
2	Samples cycled into and out of salt water (24hrs in &24 hrs out) for the 90 days immediately after casting
3	Samples cycled into and out of salt water (24hrs in &24 hrs out) for the 180 days immediately after casting
4	Samples water cured for 7 days then cycled into and out of salt water for a further 83 days
5	Samples water cured for 56 days and then constantly immersed in salt water for 28 days (ie Most Common Form of Diffusion Test)

4.TEST RESULTS

The results of all this testing are shown in the laboratory test reports in appendix A.

4.1 Physical Properties

The results of the physical testing of the concrete samples are summarised in table 4.

Table 4 - Results of Physical Tests on Concrete from Trial Mixes

Test	***Result***		
	Mix 1 Control	***Mix 2 Pore Blocker***	***Mix 3 Xypex***
Comp Strength (MPA) - 7 day	29.0	31.0	37.0
- 28 day	37.5	36.0	41.0
- 56 day	40.5	40.0	44.0
Flex Strength (MPa) - 28 day	5.3	4.6	5.8
- 56 day	5.5	4.9	5.0
Drying Shrinkage - 7 day	130	140	140
(Microstrain) - 14 day	210	190	220
- 21 day	250	250	270
- 28 day	300	290	320
- 56 day	400	430	420

4.2 Chloride Ion Penetration

The results of the chloride ion penetration testing are shown in two ways. Firstly, the actual chloride ion profiles are shown in the graphs in figures 1 to 4 following the text of this report. These chloride ion test results represented by their profiles have been used to calculate chloride ion diffusion rates, and these are shown in table 5.

Table 5 - Chloride Ion Diffusion Rates for Concrete From Trial Mixes

Curing Regime (Ref. Table 3)	*Chloride Diffusion ($m^2/s \times 10^{-12}$)*		
	Mix 1 Control	*Mix 2 Pore Blocker*	*Mix 3 Xypex*
1	13.9	11.6	5.9
2	4.9	4.5	5.9
3	5.2	4.0	5.1
4	8.9	7.9	5.8
5	11.1	16.4	8.1

5. DISCUSSION

Chloride ion penetration into concrete, and the subsequent diffusion process is a major cause of deterioration of reinforced concrete. The development of methods to slow the rate of diffusion of chloride ions has been the subject of much research, and engineers and specifiers anguish over how to specify concrete for structures that are exposed to high levels of chloride ions.

It is widely accepted that silica fume will provide good resistance to the diffusion of chloride ions into concrete. Other approaches are used, and one that has recently been specified for a number of marine structures is the use of ammonium stearate based pore blocking additive. The testing covered by this report has been carried out to determine the effectiveness of Xypex Admix in reducing the rate of ingress of chloride ions into concrete.

The diffusion testing has been carried out in a non-standard manner, in that the samples have been cycled into and out of salt water, and this has been done to model tidal and splash zones in marine environments. The results from this type of testing cannot be compared directly against the published data on chloride diffusion into concrete, however direct comparisons can be made between the three mixes that have been used in this study. Further, each mix has been subjected to a typical chloride ion diffusion test where the samples were water cured for a 56 day period, and then immersed in salt water for a period of 28 days. The results of these particular tests can be compared with the literature, however it should be noted that the water-binder ratio will have a significant bearing on the performance of the concrete. The water-binder ratio of the concretes used in these trials was 0.4, and while this is low, it would be necessary to go to a significantly lower water-binder ratio to achieve chloride diffusion rates of less than $1 m^2/s \times 10^{-12}$ (which is the level that would be required for severe exposures and long design lives).

The concrete containing Xypex has shown significant improvement over the other concretes in a typical diffusion test, and for concrete that is cured for 7 days prior to the exposure to the marine environment. These two tests are relevant to the use of a durability additive in concrete for a marine environment, for the following reasons.

- Diffusion using constant immersion is the method that is normally used in the assessment of the suitability of particular concretes for aggressive environments.

- 7 days curing and then exposure to the marine environment is a very common form of construction. Whilst curing is often difficult, specifiers of this type of project are aware of the benefits that curing can provide, and accordingly, this aspect of construction is closely scrutinised.

It is well understood that products such as slag are of significant benefit in terms of chloride ion diffusion, however these will require a minimum of 14 days curing to achieve the levels of performance that are expected. This is often difficult, and lapses in curing will result in a significant drop off in performance. Therefore, the ability to improve rates of diffusion with an alternative approach that requires a shorter period of curing could be of advantage.

The results of the testing by cycling the samples in and out of the salt water have indicated that all concretes perform in a similar manner. It is apparent that there is no significant benefit in the use of pore blocking additives in the long term with respect to chloride ion diffusion. This certainly contradicts the theory that a pore blocking additive provides immediate protection from the penetration of chloride ions, as the concrete containing this type of additive performed little better than the other concretes at any age.

As stated earlier in this report, the concretes in this series of trials were also tested to assess physical performance. From this testing, it is apparent that Xypex does not reduce the performance of concrete in any area, and it increases the strength of the concrete at all ages. The results in table 4 indicate that the flexural strength of the concrete containing Xypex has reduced between 28 and 56 days. It should be pointed out that the flexural testing was carried out on only one sample at each age, due to the late inclusion of this testing in the program. On this basis, it is expected that the 56 day Xypex result is not representative of the likely performance of concrete containing Xypex. It is certainly clear that the Xypex treated concrete has higher strength than the control mix and the pore blocking additive mix, and there is no reason that the flexural performance at 56 days should not reflect this trend.

Further testing has been carried out on samples of concrete supplied by Xypex. It is apparent on the basis of these samples that the differences between the Xypex treated concrete and other concretes in the study are greater than indicated in the samples made in this series of laboratory trials.

It is also apparent that the Xypex has no adverse effect on the drying shrinkage performance of concrete. This is very important in marine concrete, as increased drying shrinkage can lead to cracking, and cracks in concrete in a marine environment can lead to rapid durability problems. In fact, if concrete in a marine environment cracks, the work done to provide durable concrete can be un-done, as the cracks provide a rapid path to the reinforcement for the chloride ions.

6. CONCLUSION

From this testing it is apparent that concrete containing Xypex admix will have better chloride diffusion resistance than concrete made with type SL cement, particularly if the concrete is given 7 days curing. Further, Xypex treated concrete performs significantly better than both plain cement concrete and concrete containing a pore blocking additive when tested using a standard full immersion chloride ion diffusion test. This suggests that there are applications where the durability of concrete for marine applications can be enhanced by the use of Xypex admix in the concrete.

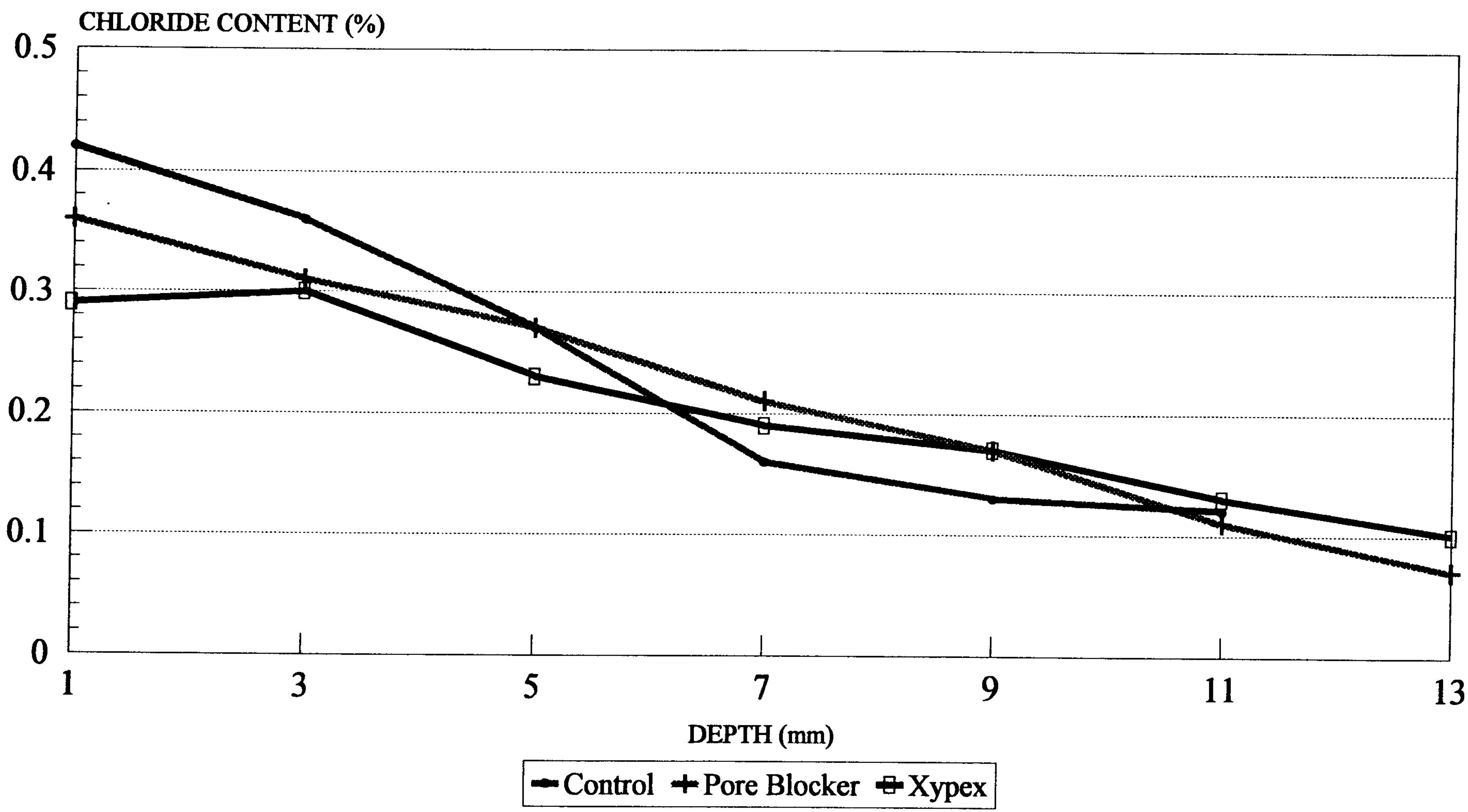
By observation of Xypex treated concrete used in the field, it is also apparent that the material has the ability to self-heal should cracking occur. This is a characteristic that is of further benefit in concrete that is exposed to aggressive environments.

D. R. Mahaffey

MAHAFFEY ASSOCIATES

CLIENT : Xypex Australia

PROJECT : Durability Trial Mixes



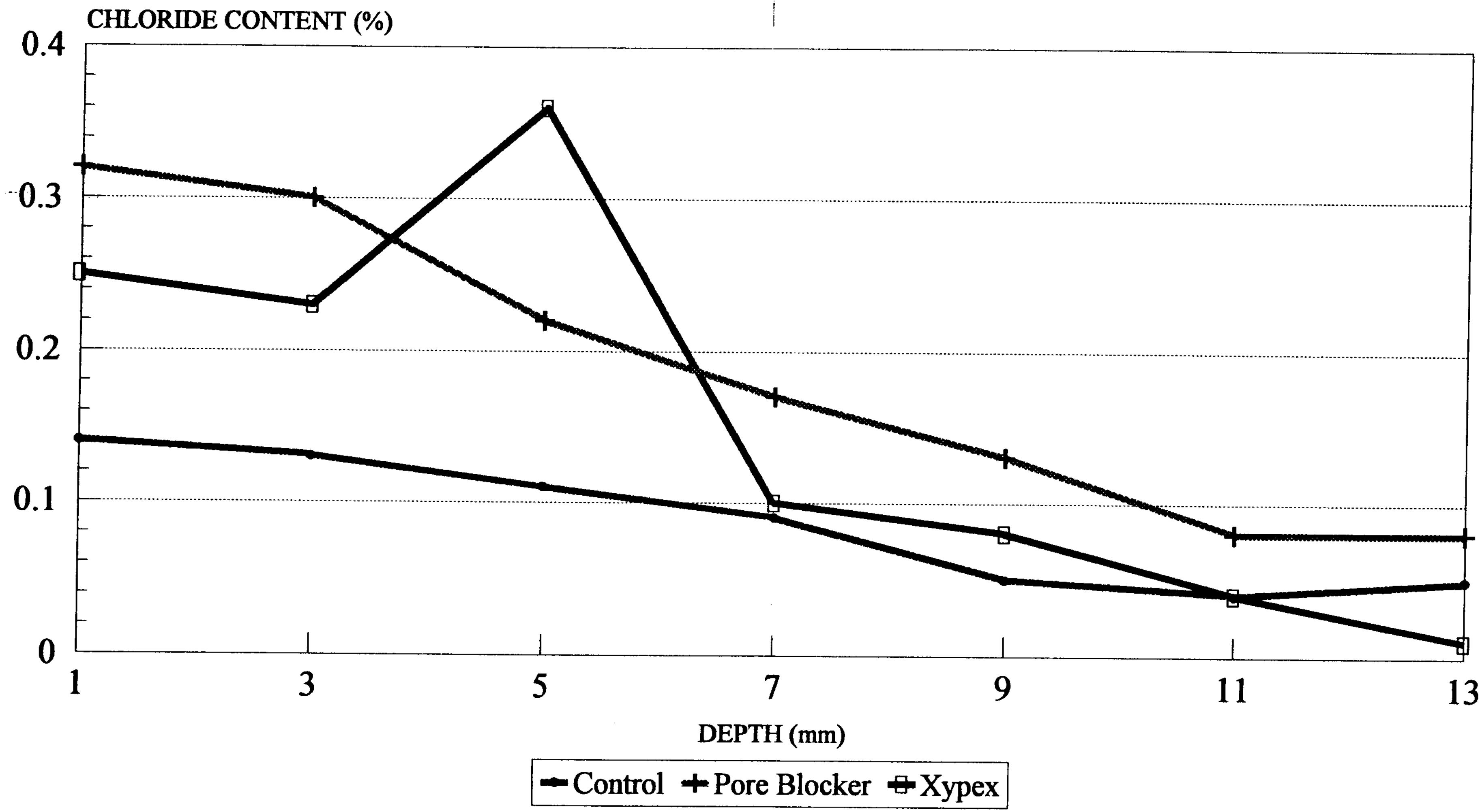
CHLORIDE PROFILES AFTER 90 DAYS OF CYCLED IMMERSION IN MARINE WATER

XYPEX3.309

MAHAFFEY ASSOCIATES

CLIENT : Xypex Australia

PROJECT : Durability Trial Mixes



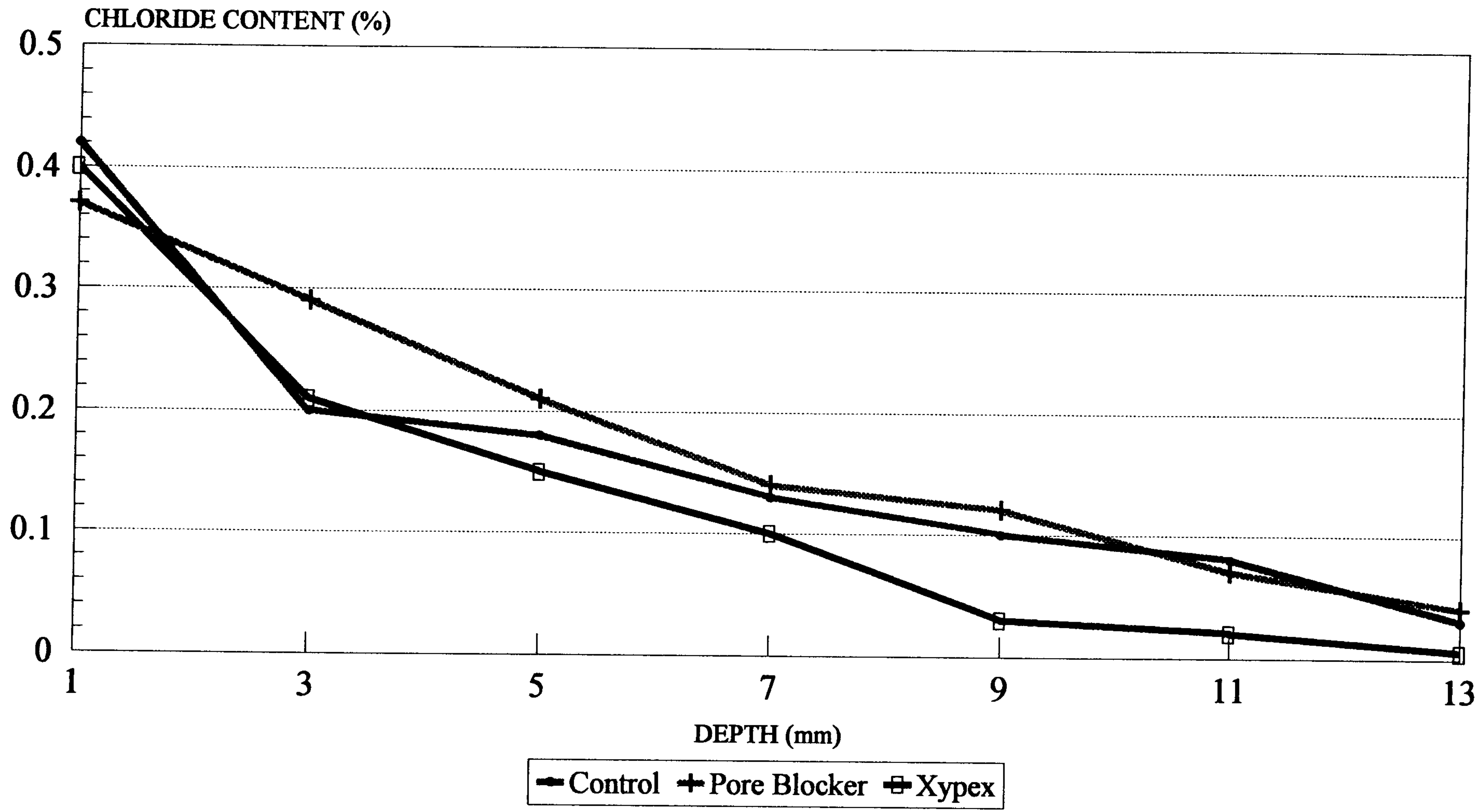
CHLORIDE PROFILES AFTER 56 DAYS OF WATER CURING AND THEN 28 DAYS CONSTANT IMMERSION IN SALT WATER

XYPEX2.309

MAHAFFEY ASSOCIATES

CLIENT : Xypex Australia

PROJECT : Durability Trial Mixes



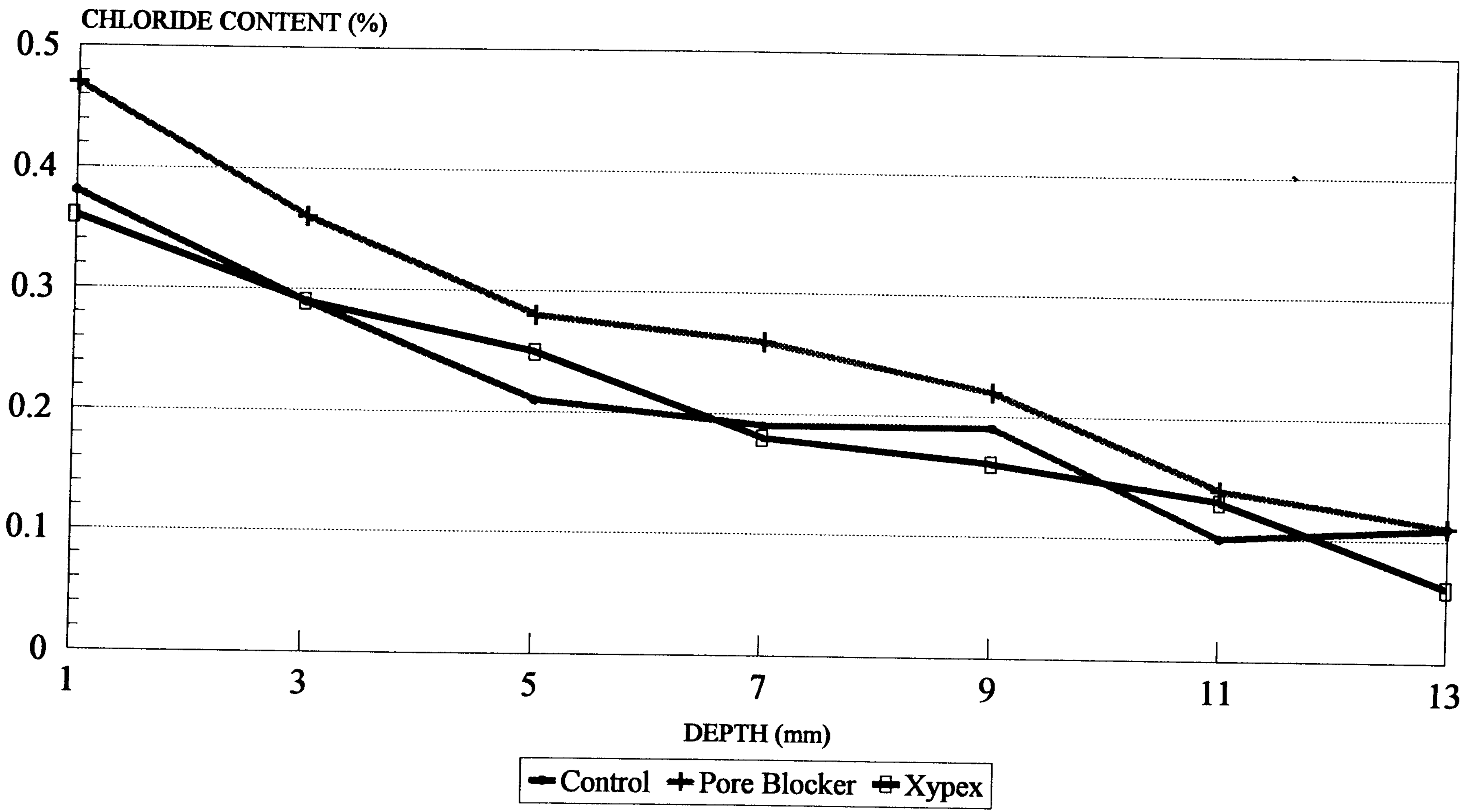
CHLORIDE PROFILES AFTER 28 DAYS OF CYCLED IMMERSION IN MARINE WATER

XYPEX1.309

MAHAFFEY ASSOCIATES

CLIENT : Xypex Australia

PROJECT : Durability Trial Mixes



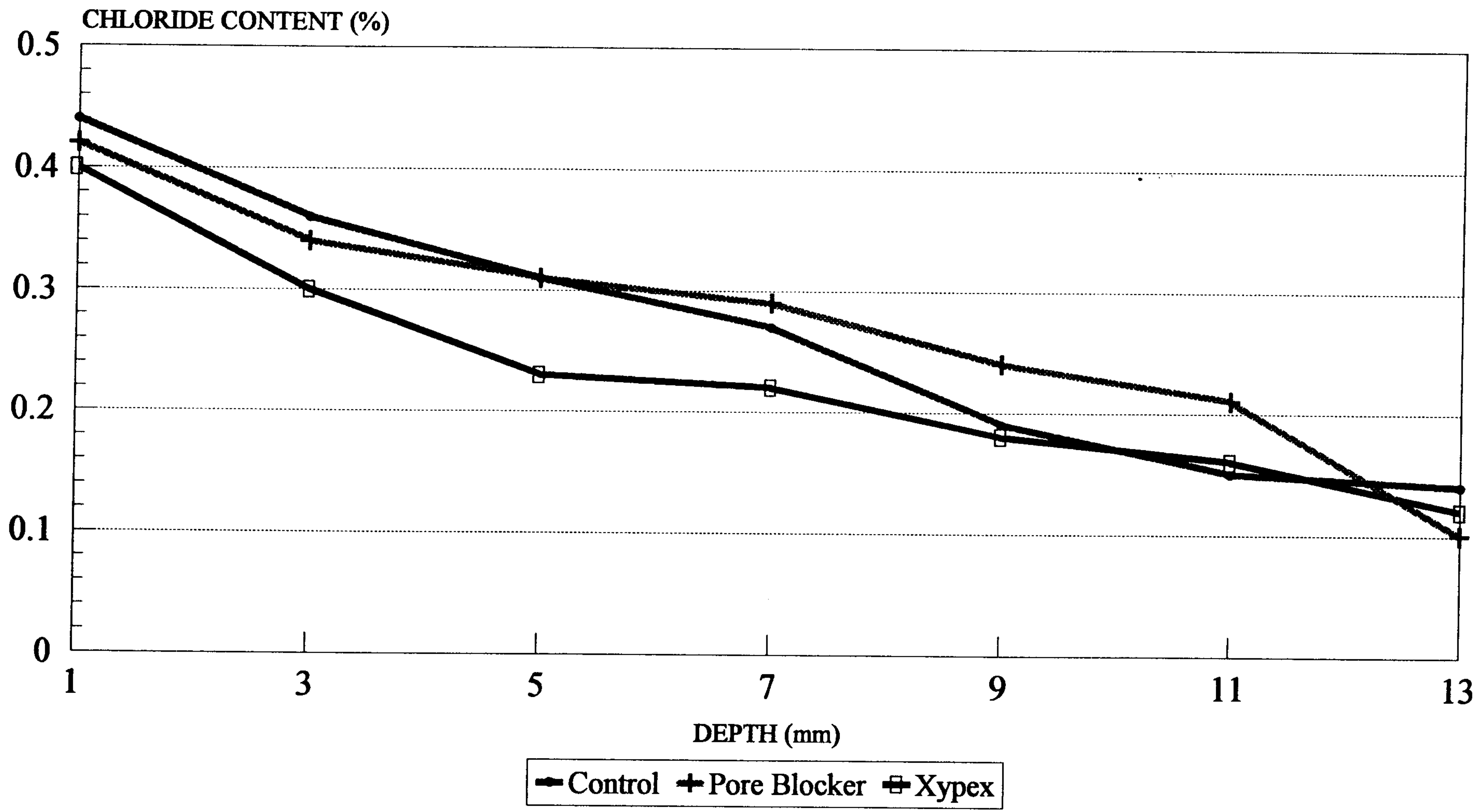
CHLORIDE PROFILES AFTER 7 DAYS OF WATER CURING THEN 83 DAYS OF CYCLED IMMERSION IN MARINE WATER

XYPEX4.309

MAHAFFEY ASSOCIATES

CLIENT : Xypex Australia

PROJECT : Durability Trial Mixes



CHLORIDE PROFILES AFTER 180 DAYS OF CYCLED IMMERSION IN MARINE WATER

XYPEXS.309

APPENDIX A

Test Results

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L07181

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM1 - 180 day

DATE : 01-04-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.44
2-4mm	0.36
4-6mm	0.31
6-8mm	0.27
8-10mm	0.19
10-12mm	0.15
12-14mm	0.14
14-16mm	0.13

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L07182

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM2 - 180 day

DATE : 01-04-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.42
2-4mm	0.34
4-6mm	0.31
6-8mm	0.29
8-10mm	0.24
10-12mm	0.21
12-14mm	0.10
14-16mm	0.10

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L07184

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM4 - 180 day

DATE : 01-04-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.40
2-4mm	0.30
4-6mm	0.23
6-8mm	0.22
8-10mm	0.18
10-12mm	0.16
12-14mm	0.12
14-16mm	0.10

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06520

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM1 - 28 Days

DATE : 13-11-95

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.42
2-4mm	0.20
4-6mm	0.18
6-8mm	0.13
8-10mm	0.10
10-12mm	0.08
12-14mm	0.03

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06428

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM2 - 28 Day

DATE : 19-10-95

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.37
2-4mm	0.29
4-6mm	0.21
6-8mm	0.14
8-10mm	0.12
10-12mm	0.07
12-14mm	0.04

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06465

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trials TM4 - 28 Day

DATE : 02-11-95

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.40
2-4mm	0.21
4-6mm	0.15
6-8mm	0.10
8-10mm	0.03
10-12mm	0.02
12-14mm	< 0.01
14-16mm	< 0.01

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06709

TEST REPORT

CLIENT : Xypex Australia Pty Ltd

SITE : Durability Trial TM1 - 90 Days

DATE : 19-12-95

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.42
2-4mm	0.36
4-6mm	0.27
6-8mm	0.16
8-10mm	0.13
10-12mm	0.12

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06888

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM2 - 90 Days

DATE : 25-01-96

MATERIAL : Concrete Cylinders

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.36
2-4mm	0.31
4-6mm	0.27
6-8mm	0.21
8-10mm	0.17
10-12mm	0.11
12-14mm	0.07
14-16mm	0.02

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06885

TEST REPORT

CLIENT : Xypex Australia

SITE : Durability Trial TM4 - 90 Days

DATE : 25-01-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.29
2-4mm	0.30
4-6mm	0.23
6-8mm	0.19
8-10mm	0.17
10-12mm	0.13
12-14mm	0.10
14-16mm	0.03

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06889

TEST REPORT

CLIENT : Xypex Australia

SITE : TM1-56day Std + 28day Salt Cur   DATE : 25-01-96

MATERIAL : Concrete Cylinder   JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.14
2-4mm	0.13
4-6mm	0.11
6-8mm	0.09
8-10mm	0.05
10-12mm	0.04
12-14mm	0.05
14-16mm	< 0.01

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06740

TEST REPORT

CLIENT : Xypex (Australia) Pty Ltd

SITE : TM2-56day Std+28day Salt Cur. DATE : 22-12-95

MATERIAL : Concrete Cylinder JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.32
2-4mm	0.30
4-6mm	0.22
6-8mm	0.17
8-10mm	0.13
10-12mm	0.08
12-14mm	0.08

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06884

TEST REPORT

CLIENT : Xypex Australia

SITE : TM4-56day Std + 28day Salt Cur

DATE : 25-01-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.25
2-4mm	0.23
4-6mm	0.36
6-8mm	0.10
8-10mm	0.08
10-12mm	0.04
12-14mm	0.01
14-16mm	< 0.01

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06739

TEST REPORT

CLIENT : Xypex (Australia) Pty Ltd

SITE : TM1-90Day,Initial 7Day Std Cur

DATE : 22-12-95

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.38
2-4mm	0.29
4-6mm	0.21
6-8mm	0.19
8-10mm	0.19
10-12mm	0.10
12-14mm	0.11

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

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L06873

TEST REPORT

CLIENT : Xypex Australia

SITE : TM2-90Day Initial 7Day Std Cur

DATE : 23-01-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.47
2-4mm	0.36
4-6mm	0.28
6-8mm	0.26
8-10mm	0.22
10-12mm	0.14
12-14mm	0.11

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

D. J. Wilmshurst

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L06875

TEST REPORT

CLIENT : Xypex Australia

SITE : TM4-90Day Initial 7Day Std Cur

DATE : 23-01-96

MATERIAL : Concrete Cylinder

JOB No. 4821

RE : CHLORIDE ION CONTENT

SAMPLE No	CHLORIDE ION CONTENT %
0-2mm	0.36
2-4mm	0.29
4-6mm	0.25
6-8mm	0.18
8-10mm	0.16
10-12mm	0.13
12-14mm	0.06

Results calculated to the nearest 0.01 %

Sampled by Bemac.

Tested in accordance with AS 1012.20, using the Volhard Method.

D. J. Wilmshurst

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TEST REPORT

This Laboratory is registered by the National Association of Testing Authorities, Australia. The tests reported herein have been performed in accordance with its terms of registration.

CLIENT: Xypex Australia

JOB: Durability Trials

SECTION: Trial Mix 1

DATE CAST: 06-09-95

JOB No. 4821

L06334

REFERENCE NUMBER CLIENT	REFERENCE NUMBER BEMAC	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	DIAMETER (mm)	LENGTH (mm)	COMPRESSIVE STRENGTH (MPa)	MASS PER UNIT VOLUME - SSD (kg m^{-3})
A	19452	04-10-95	28	27	100	199	37.0	2360
B	19453	04-10-95	28	27	101	200	38.0	2320

Samples cast by BEMAC.
Tested in accordance with AS 1012 Parts 9, and 12 (Method 1)
Cured in the laboratory in accordance with AS 1012 part 8 (Standard Temperate Zone)
Strength results calculated to the nearest 0.5 MPa.
Rubber capped.

SIGNED: [signature]

D. J. Wilmshurst.
17th October, 1995

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TEST REPORT

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CLIENT: Xypex Australia

JOB: Durability Trials

DATE CAST: 06/09/95

SECTION: Trial Mix 1

JOB No. 4821

L06493

REFERENCE NUMBER CLIENT	REFERENCE NUMBER BEMAC	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	DIAMETER (mm)	LENGTH (mm)	COMPRESSIVE STRENGTH (MPa)	MASS PER UNIT VOLUME - SSD (kg m^{-3})
A	19454	01/11/95	56	55	100	199	36.5	2380
B	19455	01/11/95	56	55	100	199	44.0	2360

Samples cast by BEMAC.
Tested in accordance with AS 1012 Parts 9, and 12 (Method 1)
Cured in the laboratory in accordance with AS 1012 part 8 (Standard Temperate Zone)
Strength results calculated to the nearest 0.5 MPa.
Rubber capped.

SIGNED: [signature]
D. J. Wilmshurst.
7th November, 1995

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TEST REPORT

CLIENT: Xypex Australia

JOB: Durability Trials

SECTION: Trial Mix 1

DATE CAST: 06-09-95

JOB No. 4821

L06335

REF No. CLIENT	BEMAC	DATE REC'D	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	MODULUS OF RUPTURE (MPa)	MASS PER UNIT OF VOLUME - SSD (kg m⁻³)
	19456	07-09-95	04-10-95	28	27	5.3	2340

Samples Cast by Bemac.
Tested in accordance with AS 1012 Parts 11 and 12 (Method 1).
Strength results calculated to the nearest 0.1 MPa
Sample size - 350* 100* 100mm

SIGNED:

D. J. Wilmshurst.
6th October, 1995

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TEST REPORT

CLIENT: Xypex Australia

JOB: Durability Trials

SECTION: Trial Mix 1

DATE CAST: 06/09/95

JOB No. 4821

L06494

REF No. CLIENT	BEMAC	DATE REC'D	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	MODULUS OF RUPTURE (MPa)	MASS PER UNIT OF VOLUME - SSD (kg m^{-3})
	19457	07/09/95	01/11/95	56	55	5.5	2460

Samples Cast by Bemac.
Tested in accordance with AS 1012 Parts 11 and 12 (Method 1).
Strength results calculated to the nearest 0.1 MPa
Sample size - 350* 100* 100mm

SIGNED: [signature]
D. J. Wilmshurst.
7th November, 1995

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TEST REPORT

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CLIENT: Xypex Australia

JOB: Durability Trials

SECTION: Trial Mix 2

DATE CAST: 12-09-95

JOB No. 4821

L06358

REFERENCE NUMBER CLIENT	REFERENCE NUMBER BEMAC	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	DIAMETER (mm)	LENGTH (mm)	COMPRESSIVE STRENGTH (MPa)	MASS PER UNIT VOLUME - SSD ($kg\ m^{-3}$)
A	19481	10-10-95	28	26	100	199	35.0	2360
B	19482	10-10-95	28	26	100	199	37.0	2380

Samples cast by BEMAC.
Tested in accordance with AS 1012 Parts 9, and 12 (Method 1)
Cured in the laboratory in accordance with AS 1012 part 8 (Standard Temperate Zone)
Strength results calculated to the nearest 0.5 MPa.
Rubber capped.

SIGNED:

D. J. Wilmshurst.
10th October, 1995

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This Laboratory is registered by the National Association of Testing Authorities, Australia. The tests reported herein have been performed in accordance with its terms of registration.

TEST REPORT

CLIENT: Xypex Australia
JOB: Durability Trials
SECTION: Trial Mix 2

DATE CAST: 12/09/95
JOB No. 4821

L06503

REFERENCE NUMBER CLIENT	REFERENCE NUMBER BEMAC	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	DIAMETER (mm)	LENGTH (mm)	COMPRESSIVE STRENGTH (MPa)	MASS PER UNIT VOLUME - SSD (kg m⁻³)
A	19483	07/11/95	56	54	100	198	40.0	2380
B	19484	07/11/95	56	54	100	198	40.0	2360

Samples cast by BEMAC.
Tested in accordance with AS 1012 Parts 9, and 12 (Method 1)
Cured in the laboratory in accordance with AS 1012 part 8 (Standard Temperate Zone)
Strength results calculated to the nearest 0.5 MPa.
Rubber capped.

SIGNED: [signature]
D. J. Wilmshurst.
7th November, 1995

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TEST REPORT

CLIENT: Xypex Australia

JOB: Durability Trials

SECTION: Trial Mix 2

JOB No. 4821

L06561

REF No. CLIENT	BEMAC	DATE CAST	DATE DEMOULDED	DATE OF INITIAL MEASUREMENT	DURATION OF STANDARD MOIST CURING (Days)	DRYING SHRINKAGE (MICROSTRAIN) 7 Days	14 Days	21 Days	28 Days	56 Days
A	19487	12-09-95	14-09-95	19-09-95	5	120	176	244	280	416
B	19488	12-09-95	14-09-95	19-09-95	5	148	184	260	304	444
C	19489	12-09-95	14-09-95	19-09-95	5	144	204	260	296	436
					AVERAGE =	140	190	250	290	430

Samples Cast by Bemac. Concrete sampled and moulded in the laboratory.
Tested in accordance with AS 1012 Part 13 - 1992.

SIGNED: [signature]

D. J. Wilmshurst
16th November, 1995

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TEST REPORT

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CLIENT: Xypex Australia
JOB: Durability Trials
SECTION: Trial Mix 2

DATE CAST: 12/09/95
JOB No. 4821

L06495

REF No. CLIENT	REF No. BEMAC	DATE REC'D	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	MODULUS OF RUPTURE (MPa)	MASS PER UNIT OF VOLUME - SSD ($kg\ m^{-3}$)
	19486	14/09/95	07/11/95	56	54	4.9	2380

Samples Cast by Bemac.
Tested in accordance with AS 1012 Parts 11 and 12 (Method 1).
Strength results calculated to the nearest 0.1 MPa
Sample size - 350* 100* 100mm

SIGNED:

D. J. Wilmshurst.
7th November, 1995

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TEST REPORT

This Laboratory is registered by the National Association of Testing Authorities, Australia. The tests reported herein have been performed in accordance with its terms of registration.

NATA

CLIENT: Xypex Australia

JOB: Durability Trials

SECTION: Trial Mix 2

DATE CAST: 12-09-95

JOB No. 4821

L06359

REF No. CLIENT	BEMAC	DATE REC'D	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	MODULUS OF RUPTURE (MPa)	MASS PER UNIT OF VOLUME - SSD (kg m^{-3})
	19485	14-09-95	10-10-95	28	26	4.6	2360

Samples Cast by Bemac.
Tested in accordance with AS 1012 Parts 11 and 12 (Method 1).
Strength results calculated to the nearest 0.1 MPa
Sample size - 350* 100* 100mm

SIGNED: [signature]
D. J. Wilmshurst.
10th October, 1995

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TEST REPORT

CLIENT: Xypex Australia

JOB: Durability Trials — **DATE CAST:** 20/09/95

SECTION: Trial Mix 4 — **JOB No.** 4821

L06417

REFERENCE NUMBER CLIENT	REFERENCE NUMBER BEMAC	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	DIAMETER (mm)	LENGTH (mm)	COMPRESSIVE STRENGTH (MPa)	MASS PER UNIT VOLUME - SSD (kg m^{-3})
A	19492	18/10/95	28	26	99	200	41.5	2360
B	19493	18/10/95	28	26	100	200	40.5	2360

Samples cast by BEMAC.
Tested in accordance with AS 1012 Parts 9, and 12 (Method 1)
Cured in the laboratory in accordance with AS 1012 part 8 (Standard Temperate Zone)
Strength results calculated to the nearest 0.5 MPa.
Rubber capped.

SIGNED: [signature]
D. J. Wilmshurst.
24th October, 1995

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TEST REPORT

This Laboratory is registered by the National Association of Testing Authorities, Australia. The tests reported herein have been performed in accordance with its terms of registration.

CLIENT: Xypex Australia

JOB(1): Durability Trials

SECTION(2): Trial Mix 4

JOB No. 4821

L06628

REF No. CLIENT (3)	BEMAC	DATE CAST (4)	DATE REC'D	DATE OF INITIAL MEASUREMENT	DURATION OF STANDARD MOIST CURING (Days)	DRYING SHRINKAGE (MICROSTRAIN) 7 Days	14 Days	21 Days	28 Days	56 Days
A	20248	20-09-95	22-09-95	27-09-95	5	140	372	436	480	584
B	20249	20-09-95	22-09-95	27-09-95	5	128	208	260	308	408
C	20250	20-09-95	22-09-95	27-09-95	5	140	228	288	336	440
					AVERAGE =	140	220	270	320	420

Samples Cast by Others. Information supplied by client denoted as follows: (1),(2),(3),(4)
Tested in accordance with AS 1012 Part 13 - 1992.
The average drying shrinkage results are based on individual results within 40 microstrain of the median, as required by clause 8d of the standard.

SIGNED:

D. J. Wilmshurst
5th December, 1995

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TEST REPORT

CLIENT: Xypex Australia
JOB: Durability Trials
SECTION: Trial Mix 4

DATE CAST: 20-09-95
JOB No. 4821

L06570

REFERENCE NUMBER CLIENT	REFERENCE NUMBER BEMAC	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	DIAMETER (mm)	LENGTH (mm)	COMPRESSIVE STRENGTH (MPa)	MASS PER UNIT VOLUME - SSD ($kg\ m^{-3}$)
A	19494	15-11-95	56	54	100	200	43.5	2360
B	19495	15-11-95	56	54	100	200	44.5	2380

Samples cast by BEMAC.
Tested in accordance with AS 1012 Parts 9, and 12 (Method 1)
Cured in the laboratory in accordance with AS 1012 part 8 (Standard Temperate Zone)
Strength results calculated to the nearest 0.5 MPa.
Rubber capped.

SIGNED: [signature]
D. J. Wilmshurst.
21st November, 1995

BEMAC LABORATORIES
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TEST REPORT

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[P.O. Box 349]
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Phone: (02) 684 2422
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CLIENT: Xypex Australia

JOB: Durability Trials — DATE CAST: 20/09/95

SECTION: Trial Mix 4 — JOB No. 4821

L06418

REF No. CLIENT	BEMAC	DATE REC'D	DATE TESTED	AGE TESTED (Days)	DURATION OF STANDARD CURING (Days)	MODULUS OF RUPTURE (MPa)	MASS PER UNIT OF VOLUME - SSD (kg m^{-3})
	19497	22/09/95	18/10/95	28	26	5.8	2360

Samples Cast by Bemac.
Tested in accordance with AS 1012 Parts 11 and 12 (Method 1).
Strength results calculated to the nearest 0.1 MPa
Sample size - 350* 100* 100mm

SIGNED: [signature]
D. J. Wilmshurst.
24th October, 1995

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Phone: 02 9684 2422 Fax: 02 9684 2310

Report on

CHLORIDE ION PENETRATION INTO CONCRETE CONTAINING XYPEX

Report No : MR431

Date : March 1996

Client : Xypex Australia

1. BRIEF

To carry out a test program on Xypex treated concrete to determine the effects on chloride ion diffusion characteristics that may result from the use of Xypex as an admixture.

To determine if early pore blocking in concrete will have any long term benefits in terms of durability.

To assess a number of other physical properties of concrete containing Xypex Admix, and to compare these with other concretes. These are -

- Compressive Strength
- Drying Shrinkage
- Flexural Strength

2. APPROACH

A series of comparative trials was deemed to be the best approach in determining the performance, and a number of concretes with known performance could be included in the trial series.

A series of three trials was ultimately chosen, as follows.

1. Control mix - Portland cement only, and no waterproofing additives
2. Pore Blocking additive - Portland cement only, with a pore blocking additive, based on ammonium sterate.
3. Xypex - Portland cement only with Xypex Admix

Samples were cast from these mixes, for testing to determine the resistance to chloride ion diffusion. This was done under a number of conditions, in an effort to model actual field conditions. To this end, diffusion samples were subjected to a series of cycles of immersion in salt water, followed by a period in air.

This approach is not normally taken in the assessment of chloride ion diffusion, as this characteristic is normally determined after a period of constant immersion. Whilst this provides a constant condition for the purposes of comparison, it does not indicate the type of performance may be achieved in, say, a tidal area. Various intervals were used in this testing, to ensure that performance with time could be determined.

To allow the relative performance of the Xypex treated concrete to be assessed against the recognised standard, a series of tests was carried out using constant immersion rather than cycling.

The most likely scenario in concrete that has to be protected in a marine environment is that it will receive an amount of curing (although usually not for a very long period), and then it will be exposed to the prevailing conditions. This will often mean a rising tide, when the concrete is still very young.

Durability is of increasing importance at present, and designers are looking for materials that will enhance durability performance. Accordingly, it has been suggested that pore blocking additives will be beneficial in enhancing the durability performance of concrete, because they will stop chloride ions entering (or moving through) the concrete at an early age (ie 2-3 days). Therefore the cycling process was, in some cases, commenced on stripping of the concrete, to model the likely conditions that the concrete would have faced had it been placed in a tidal zone.

A number of other properties of the various concretes would be determined, so that the possibility of the additives effecting performance in other areas could be determined.

3. TESTING

3.1 Trial Mixes

A series of three trial mixes was carried out in the laboratory, with the only variable being the durability additive that was used in the concrete.

The three mixes are shown in table 1, and it is clear from this that the binder contents, water-binder ratios and workabilities are all the same. Therefore relative performance of the additives can be assessed by making direct comparisons between the mixes.

Table 1 - Trial Mix Details

Mix	*1*	*2*	*3*
20mm (kg/m^3)	720	730	720
10mm (kg/m^3)	310	310	310
Coarse Sand (kg/m^3)	580	585	580
Fine Sand (kg/m^3)	195	200	195
Type SL Cement (kg/m^3)	365	370	365
Xypex (kg/m^3)	-	-	2.9
Pore Blocker (l/m^3)*	-	30	-

Superplasticiser (l/m^3)	1.0	1.2	1.4
Air Entrainer (ml/m^3)	100	100	100
Water/Binder	0.4	0.4	0.4
Density (kg/m^3)	2320	2330	2320
Air Content (%)	4.9	3.8	4.9
Slump (mm)	125	125	120

* This material is approx.70% water, and this water has been included in the free water used in calculating the water-binder ratio.

The mixes were made with commercially available materials, including Nepean aggregates and Type SL cement from a major supplier to the Sydney market.

3.2 Testing Regime

A series of samples was made from each mix, as set out in table 2. This also shows the test program.

Table 2 - Test Samples and Testing Regime

Samples	*Testing*
2-100mm dia. Cylinders	Compression testing at 7 days

2-100mm dia. Cylinders	**Compression testing at 28 days**
2-100mm dia. Cylinders	**Compression testing at 56 days**
1-100 x 350mm Beam	**Flexural Strength at 28 days**
1-100 x 350mm Beam	**Flexural strength at 56 days**
3-75mm Shrinkage Prisms	**Drying Shrinkage to 56 days**
5-150mmdia Cylinder	**Chloride Penetration testing at various ages using various exposures**

3.3 Chloride Penetration Testing

The testing for chloride ion penetration was carried out by immersing the samples into a bath of water maintained at a chloride ion content of 1.75%. The samples were each exposed to a range of cycles of immersion in and removal from the salt water bath, as described in table 3.

At the completion of the immersion cycles, the samples were face ground to remove increments (each of 2mm depth) of concrete dust. These dust samples were then tested for chloride ion content. This allowed the chloride ion profile of the samples to be plotted, and the diffusion rates of chloride ions into the concrete to be calculated under the various conditions.

Table 3 - Chloride Penetration Exposure Conditions

Sample	*Exposure Condition*
1	**Samples cycled into and out of salt water (24hrs in &24 hrs out) for the 28 days immediately after casting**
2	**Samples cycled into and out of salt water (24hrs in &24 hrs out) for the 90 days immediately after casting**
3	**Samples cycled into and out of salt water (24hrs in &24 hrs out) for the 180 days immediately after casting**
4	**Samples water cured for 7 days then cycled into and out of salt water for a further 83 days**
5	**Samples water cured for 56 days and then constantly immersed in salt water for 28 days (ie Most Common Form of Diffusion Test)**

4.TEST RESULTS

The results of all this testing are shown in the laboratory test reports in appendix A.

4.1 Physical Properties

The results of the physical testing of the concrete samples are summarised in table 4.

Table 4 - Results of Physical Tests on Concrete from Trial Mixes

Test	*Result*		
	Mix 1 *Control*	*Mix 2* *Pore Blocker*	*Mix 3* *Xypex*
Comp Strength (MPA) - 7 day	**29.0**	**31.0**	**37.0**
- 28 day	**37.5**	**36.0**	**41.0**
- 56 day	**40.5**	**40.0**	**44.0**
Flex Strength (MPa) - 28 day	**5.3**	**4.6**	**5.8**
- 56 day	**5.5**	**4.9**	**5.0**
Drying Shrinkage - 7 day	**130**	**140**	**140**
(Microstrain) - 14 day	**210**	**190**	**220**
- 21 day	**250**	**250**	**270**
- 28 day	**300**	**290**	**320**
- 56 day	**400**	**430**	**420**

4.2 Chloride Ion Penetration

The results of the chloride ion penetration testing are shown in two ways. Firstly, the actual chloride ion profiles are shown in the graphs in figures 1 to 4 following the text of this report. These chloride ion test results represented by their profiles have been used to calculate

chloride ion diffusion rates, and these are shown in table 5.

Table 5 - Chloride Ion Diffusion Rates for Concrete From Trial Mixes

Curing Regime (Ref. Table 3)	*Chloride Diffusion ($m^2/s \times 10^{-12}$)*		
	Mix 1 Control	*Mix 2 Pore Blocker*	*Mix 3 Xypex*
1	13.9	11.6	5.9
2	4.9	4.5	5.9
3	5.2	4.0	5.1
4	8.9	7.9	5.8
5	11.1	16.4	8.1

5. DISCUSSION

Chloride ion penetration into concrete, and the subsequent diffusion process is a major cause of deterioration of reinforced concrete. The development of methods to slow the rate of diffusion of chloride ions has been the subject of much research, and engineers and specifiers anguish over how to specify concrete for structures that are exposed to high levels of chloride ions.

It is widely accepted that silica fume will provide good resistance to the diffusion of chloride ions into concrete. Other approaches are used, and one that has recently been specified for a number of marine structures is the use of ammonium stearate based pore blocking additive. The testing covered by this report has been carried out to determine the effectiveness of Xypex Admix in reducing the rate of ingress of chloride ions into concrete.

The diffusion testing has been carried out in a non-standard manner, in that the samples have been cycled into and out of salt water, and this has been done to model tidal and splash zones in marine environments. The results from this type of testing cannot be compared directly against the published data on chloride diffusion into concrete, however direct comparisons can be made between the three mixes that have been used in this study. Further, each mix has been subjected to a typical chloride ion diffusion test where the samples were water cured for a 56 day period, and then immersed in salt water for a period of 28 days. The results of these particular tests can be compared with the literature, however it should be noted that the water-binder ratio will have a significant bearing on the performance of the concrete. The water-binder ratio of the concretes used in these trials was 0.4, and while this is low, it would be necessary to go to a significantly lower water-binder ratio to achieve chloride diffusion rates of less than $1m^2/s \times 10^{-12}$ (which is the level that would be required for severe exposures and long design lives).

The concrete containing Xypex has shown significant improvement over the other concretes in a typical diffusion test, and for concrete that is cured for 7 days prior to the exposure to the marine environment. These two tests are relevant to the use of a durability additive in concrete for a marine environment, for the following reasons.

- Diffusion using constant immersion is the method that is normally used in the assessment of the suitability of particular concretes for aggressive environments.
- 7 days curing and then exposure to the marine environment is a very common form of construction. Whilst curing is often difficult, specifiers of this type of project are aware of the benefits that curing can provide, and accordingly, this aspect of construction is closely scrutinised.

It is well understood that products such as slag are of significant benefit in terms of chloride ion diffusion, however these will require a minimum of 14 days curing to achieve the levels of performance that are expected. This is often difficult, and lapses in curing will result in a significant drop off in performance. Therefore, the ability to improve rates of diffusion with an alternative approach that requires a shorter period of curing could be of advantage.

The results of the testing by cycling the samples in and out of the salt water have indicated that all concretes perform in a similar manner. It is apparent that there is no significant benefit in the use of pore blocking additives in the long term with respect to chloride ion diffusion. This certainly contradicts the theory that a pore blocking additive provides immediate protection from the penetration of chloride ions, as the concrete containing this type of additive performed little better than the other concretes at any age.

As stated earlier in this report, the concretes in this series of trials were also tested to assess physical performance. From this testing, it is apparent that Xypex does not reduce the performance of concrete in any area, and it increases the strength of the concrete at all ages. The results in table 4 indicate that the flexural strength of the concrete containing Xypex has reduced between 28 and 56 days. It should be pointed out that the flexural testing was carried out on only one sample at each age, due to the late inclusion of this testing in the program. On this basis, it is expected that the 56 day Xypex result is not representative of the likely performance of concrete containing Xypex. It is certainly clear that the Xypex treated concrete has higher strength than the control mix and the pore blocking additive mix, and there is no reason that the flexural performance at 56 days should not reflect this trend.

Further testing has been carried out on samples of concrete supplied by Xypex. It is apparent on the basis of these samples that the differences between the Xypex treated concrete and other concretes in the study are greater than indicated in the samples made in this series of laboratory trials.

It is also apparent that the Xypex has no adverse effect on the drying shrinkage performance of concrete. This is very important in marine concrete, as increased drying shrinkage can lead to cracking, and cracks in concrete in a marine environment can lead to rapid durability problems. In fact, if concrete in a marine environment cracks, the work done to provide durable concrete can be un-done, as the cracks provide a rapid path to the reinforcement for the chloride ions.

6.CONCLUSION

From this testing it is apparent that concrete containing Xypex admix will have better chloride diffusion resistance than concrete made with type SL cement, particularly if the concrete is given 7 days curing. Further, Xypex treated concrete performs significantly better than both plain cement concrete and concrete containing a pore blocking additive when tested using a standard full immersion chloride ion diffusion test. This suggests that there are applications where the durability of concrete for marine applications can be enhanced by the use of Xypex admix in the concrete.

By observation of Xypex treated concrete used in the field, it is also apparent that the material has the ability to self-heal should cracking occur. This is a characteristic that is of further benefit in concrete that is exposed to aggressive environments.

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