

FIELD TESTING OF WOOD PRESERVATIVES IN CANADA VI: L-JOINT TESTING OF MILLWORK PRESERVATIVES

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Summary

Since the withdrawal of the registration of pentachlorophenol for millwork applications in Canada there has been interest in restoring a broader range of available active ingredients. To assist in this process, Forintek initiated a series of field exposure tests of millwork preservatives in 1990. Some of this work was done under contract to chemical suppliers and some as part of Forintek's core program. The method used was the L-joint test. It was designed to simulate one of the lower joints of the opening section of a window; however, the test method increases the potential for water trapping creating a somewhat accelerated test. This paper summarises recent results from some of the longest running tests.

After five years in test, ponderosa pine samples treated with 1.0% TCMTB or TCMTB/IPBC (50:50) formulations in mineral spirits remained in excellent condition when treated by 3-minute dip or double vacuum. Both formulations performed as well as a former standard preservative for millwork, 5.0% pentachlorophenol. Similarly, after six years, 1.0% propiconazole and 3.0% zinc neodecanoate gave equivalent performance to 5.0% pentachlorophenol when applied by double-vacuum. TBTO also gave equivalent performance to 5.0% pentachlorophenol when both were applied by dip treatment. Surprisingly, TBTO at 1.0% by double vacuum did not perform quite as well as 5.0% pentachlorophenol in this experiment. Solvent-only treatments showed extensive decay.

Hem-fir L-joints that had been diffusion-treated with borate were completely free from decay after six years' exposure. In contrast, untreated samples showed moderate to extensive decay. This level of protection is somewhat surprising considering that most of the borate had been lost from this material after five years.

This work suggests that a variety of preservatives could replace pentachlorophenol, a reference preservative which has provided adequate performance for millwork in the past.

Acknowledgements

Forintek Canada Corp. would like to thank its industry members, Natural Resources Canada (Canadian Forest Service), and the Provinces of British Columbia, Alberta, Ontario, Quebec, Nova Scotia and New Brunswick, for their guidance and financial support for this research.

Forintek would like to thank Buckman Laboratories International Inc. and Protim Solignum Ltd. for permission to publish results from L-joint tests carried out under contract. Forintek would also like to acknowledge the support of U.S. Borax Inc in financially supporting the chemical analysis of the borate-treated L-joints.

1. Introduction

In Canada the most commonly used preservatives for the protection of window joinery have been phenyl mercury oleate (PMO) followed by pentachlorophenol (PCP), and tributyltin oxide (TBTO). Environmental and health concerns have been expressed over the use of organomercurials and PCP, particularly where there may be frequent human contact. The use of PCP has now been discontinued. There is thus a demonstrated need for data on alternative joinery preservatives.

Most of the major population centres of North America, including Vancouver, B.C., are in the zone of medium above-ground decay potential, as calculated by Setliff (1986) using Scheffer's (1971) Climate Index. Vancouver is therefore a suitable location for field testing of experimental preservatives in protecting window joinery.

In 1990 Forintek Canada Corp. initiated L-joint tests of several millwork preservatives for a number of contract clients and for our own research purposes. One set of two light organic solvent preservatives (LOSP) was installed for Buckman Laboratories International Inc. Reference material treated with PCP was installed by Forintek. A second set of three preservatives was installed for Protim Solignum Ltd. in 1991.

In addition, a test of borate-treated millwork was set up as part of Forintek's research program. As a result of its potential as an added-value, environmentally acceptable, high-performance wood product, borate-treated lumber has attracted the interest of the Canadian forest products industry (Byrne 1990). The one factor which is likely to have the greatest impact on the performance of borate-treated millwork is the mobility of the boron. Borate-treated wood is currently approved for use in exterior exposure in New Zealand, provided it has a three-coat paint finish. Nevertheless there is concern outside New Zealand that redistribution of boron within the treated wood could lead to localized depletion and decay. The L-joint test of borate-treated wood was therefore set up mainly to determine whether preservative depletion is likely to be a problem.

This report describes the performance results from a variety of L-joint trials.

2.0 Materials and Methods

Preparation and treatment

Following the specifications of the AWWA E9-87 standard test method, ponderosa pine (*Pinus ponderosa* Laws) sapwood was machined into mortises, 38 mm square and 203 mm long, and tenons, 38 mm square and 210 mm long. Corners of the units were rounded to a radius of 2 mm. The mortises and tenons were fitted together at right angles to produce an "L-joint" or simulated window joint (Figure 1).

Buckman Laboratories Formulations

Twenty five of each unit were treated at Forintek by dip and double-vacuum processes with two experimental formulations supplied by Buckman: BL1127 (TCMTB, 2-(thiocyanomethylthio) benzothiazole) and BL1156 (50:50 TCMTB/IPBC, 3-iodo-2-propynylbutyl carbamate) at 0.15%, 0.20%, 0.50%, and 1.0% active ingredient, all in mineral spirits. In addition 25 units were treated with mineral spirits alone to act as controls.

Protim Solignum Formulations

Twenty of each unit were given one of the following four treatments by Protim Solignum Timber Treatments Division.

F955 1.0% TBTO in mineral spirits	by dip
F955 1.0% TBTO in mineral spirits	by double-vacuum
F956 1.0% propiconazole in mineral spirits	by double-vacuum
F957 3.0% (as Zn) zinc neodecanoate in mineral spirits	by double-vacuum

Forintek Reference and Borate treated Material:

As part of Forintek's field testing program, 20 units were dip- and double-vacuum-treated with 5.0% PCP in mineral spirits or mineral spirits alone. All LOSP-treated L-joints were air dried.

The borate-treated material was not treated specifically for this work because diffusion-treated hem-fir was available from an earlier study. Freshly sawn and planed nominal 2 x 4 inch green hem-fir lumber, a mixture of *Tsuga heterophylla* [Raf.] Sarg. and *Abies amabilis* [Dougl.] Forbes., had been dipped for two minutes in a 30% solution of TIM-BOR® wood preservative at 50° C. The boards were then drained for two minutes and close piled at 20° C for 12 weeks. At the end of the diffusion period the boards were kiln dried to a moisture content of 12%. The boards were machined into 30 mortises and tenons, as described above. Thirty untreated L-joints were manufactured from comparable untreated hem-fir material.

L-joint finishing

A three-coat paint finish was applied to all L-joints as follows: an exterior primer conforming to CGSB 1-GP-55M 1982 was applied at an average rate of approximately 5.5 g per unit. The first coat of alkyd enamel topcoat conforming to CGSB 1-GP-59M 1977 was applied at a rate of 5.5 g per unit and the second coat at a rate of 4.5 g per unit. Twenty-four hours was allowed for drying between coats. A cross-section about 5 mm thick was removed from the square end of each tenon for analysis. The cross-cut ends of the tenons were sealed with a flexible two-part epoxy resin containing a UV barrier, and metal identification tags were affixed to the mortise members. Each L-joint was broken apart to break the paint film and then reassembled. This was done to ensure consistency between replicates in the time to failure of the paint film at the joint.

Exposure at the test site

For the Buckman material and the reference treatments, twenty units per treatment were installed on exposure racks, in a randomized array. For the Protim Solignum material fifteen units per treatment were installed, and for the borate treatments thirty replicates were used. The tenon members were supported at a 10° angle on untreated stickers, with the mortises in contact with the back of the rack (Figure 2). The racks, originally located on the flat roof of the old Forintek Vancouver laboratory, were moved approximately 1 km. in 1991 to their current location at the rear of the new Forintek facility. They are oriented facing south.

The L-joints were refinished in 1994 with one coat of a water-based paint and the joint was again broken and reassembled.

At this test site, temperatures annually average 10° C, with a December average of 3° C and a July average of 17° C. The site receives about 1900 hours of bright sunshine and approximately 1250 mm of precipitation per year, with an average 34 mm of rain in July and 140 mm of rain in December. The climate index of the site is 46 (Setliff 1986) and it therefore falls into the intermediate hazard zone for above-ground exposure (DeGroot and Esenther 1982, Scheffer 1971).

The predominant wood rotting basidiomycete visible as fruiting bodies on L-joints at this site is *Gloeophyllum sepiarium* (Fr.) Karsten; with a *Stereum sp.*, a *Merulius sp.* and *Gloeophyllum protractum* (Fr.) Imazeki as minor species.

Inspection

To evaluate the extent of decay, each L-joint was visually inspected as an intact unit, then broken apart, and the tenon members were rated according to the AWPAs E9-87 standard (AWPA 1991). If the presence of decay was suspected, the area was gently probed for softness.

<u>Rating</u>	<u>Condition of L-joint</u>
0	Sound
1	Signs of slight surface decay
2	Small zones of obvious decay
3	Extensive decay
4	Rejected: the specimen is no longer an integral unit because of decay. As a component in practice it would have to be replaced at this point.

A mean rating was calculated for each preservative retention level.

Note: In AWPAs E9-87 the presence of staining fungi does not merit a rating of 1.

Analysis of borate distribution

The cross-sections which had been removed from the square end of each tenon prior to being put into test were analyzed for initial borate content in 1992. Each piece was divided into nine equal segments in a 3 x 3 array. Each segment was analyzed separately for borate content using hot water extraction and mannitol titration (Winters, undated).

After the five-year inspection in May 1995, five borate-treated L-joints were randomly selected for removal from test for depletion analysis. These five L-joints were allowed to dry in the laboratory for about three weeks, at which time the moisture content was determined to be 9.5%. Five mm thick slices were removed along the length of the tenon, at 0, 20, 40, 60, 80, 100, 120, and 140 mm from the shoulder of the joint. In addition a 5 mm thick slice was cut from the tenon itself, 20 mm from the shoulder. Each 38 x 38 mm slice was divided into nine sub-samples, as above. The 38 x 13 mm slice from the tenon was cut into three sub-samples. The 75 samples per L-joint were individually analyzed for borate content, as above.

Forintek has done no chemical analyses on the Buckman, Protim or Forintek reference material.

3. Results and Discussion

Retentions determined by uptake at the time of treatment are given in Tables 1 to 4 and decay ratings are given in Tables 5 to 8.

Buckman Laboratories Formulations

After five years of exposure, the L-joints treated with 1.0% TCMTB showed no signs of decay (Table 5). The majority of the 1.0% treated samples appeared to be dry in the joint area. This suggests that the water-repellent component was still contributing to the protective effect of the formulation. L-joints treated with less than 1.0% TCMTB by dip, and less than 0.5% TCMTB by double-vacuum, showed some signs of early decay, with mean decay ratings of 0.1 to 0.7.

For TCMTB/IPBC, no decay was found in joints dipped or double-vacuum treated with 1.0% treating solution. Results at the three lower retention levels were comparable to those of TCMTB (Table 5). There was little to distinguish between the performance of TCMTB and TCMTB/IPBC.

The results for both TCMTB and TCMTB/IPBC at 1.0% compared favourably to 5.0% PCP (Table 7) where there was slight decay in dip-treated joints (mean rating 0.3). No decay was found after five years in L-joints that had been double-vacuum treated with PCP. However, there was no statistically significant difference between the performance of PCP and TCMTB or TCMTB/IPBC with either dip or double-vacuum treatments, using a two-sample *t*-test ($p < 0.05$).

Overall, double-vacuum treatments greatly outperformed dip treatments. Dip treatments displayed a very abrupt threshold of decay. Any treatments containing less than 1.0% a.i. had up to three L-joints with advanced decay, rated 3. The overall decay rating of L-joints treated with 0.5% TCMTB by dip was 0.7 (slight surface decay present). The comparable set of L-joints treated by the double-vacuum process showed no decay. Table 1 illustrates that preservative active ingredient uptake was equivalent for the 1.0% TCMTB by dip and for 0.5% TCMTB by double-vacuum; i.e. double-vacuum doubled the preservative uptake.

Protim Solignum Formulations

After six years of exposure, the L-joints that had been dip- or double-vacuum-treated with 1.0% TBTO showed evidence of some decay with mean ratings of 0.2 (Table 6). Both sets had one unit at a rating of 1 and one unit at a rating of 2. This was a somewhat surprising result considering the weight of evidence showing that double-vacuum treatments are superior to dip treatments (Orsler and Smith 1993). L-joints treated with 1.0% propiconazole and

3.0% zinc neodecanoate were all completely sound (Table 6).

After seven years, decay in solvent-treated controls was moderate to extensive in both dip- and double-vacuum treated material, with mean decay ratings of 3.0 and 2.7 respectively (Table 7). The mean decay ratings of these controls after five years were higher than the comparable Buckman material (Table 5), although the difference was not statistically significant.

The units that had been dip-treated with 5.0% PCP had a mean rating of 0.6 after seven years (two rated 1, three rated 2 and one rated 4, one unit was missing), whereas the double-vacuum-treated material was all completely sound (Table 7).

The difference in the timing of installation and the timing of inspection between the experimental and reference treatments means that comparisons should be made with caution. The absence of decay in L-joints that had been double-vacuum treated with 1.0% propiconazole and 3.0% zinc neodecanoate matched the performance of PCP over similar periods. After six years, L-joints dip-treated with TBTO showed a similar mean rating to L-joints that had been dip-treated with PCP after five years' exposure.

Borate-treated hem-fir

The mean ratings for untreated hem-fir L-joints increased from 0.6 after two years to 2.7 after six years in test (Table 8). After six years, 15 of the 30 untreated L-joints had failed from decay and were rated 4. In contrast, the borate-treated L-joints have remained completely sound during the test. Due to the limited scope of this work, no hem-fir L-joints were treated with a reference preservative. However, some inferences can be drawn from comparison with an industry standard, ponderosa pine dip-treated with 5.0% PCP. This material had suffered limited decay, with a mean rating of 0.3 after five years and 0.6 after seven years (Table 7).

Initially, all the L-joints had mean borate contents above the 0.1% BAE specified for the one-ninth core of Canadian hemlock (Table 4) but only one was higher than the cross-section retention requirement of 0.4% BAE for treated lumber in BWPA standard 105 (British Wood Preserving Association 1977). Considering that the treatment method specified in BWPA 105 had been followed, these low retentions were almost certainly due to the removal of surface layers with high borate retentions by planing during manufacture of the L-joints from nominal 2 x 4 inch lumber.

After five years, the mean borate content of the 75 analyses per joint was substantially less than that found in the samples removed prior to exposure (9 analyses per joint) in all five L-joints analysed. The average residual level ranged from 16% to 39% of that found initially (Table 4). In all cases this was below the average 0.1% BAE specified in the BWPA standard

for hemlock treatment.

In four of the five L-joints examined, borate content decreased from between 0.25% and 0.35% BAE near the distal end of the tenon to 0.00% BAE towards the joint (Figures 3,5,6 and 7). In the fifth L-joint (#11) the borate content was at a maximum at 100 mm from the joint (Figure 4) and decreased both towards the tenon and towards the distal end. An examination of the distal end of L-joint #11 revealed a split in the wood which had cracked the end-seal. This would have allowed ingress of water and leaching.

In three of the L-joints, the amount of borate found at the 140 mm position was more than that found in the initial slice taken prior to exposure at approximately 170 mm from the tenon shoulder. This ratio was 118% in L-joint #23, 120% in L-joint #32, and 156% in L-joint #26. No detailed analysis was done on the distribution along the length of the L-joints prior to exposure, consequently it cannot be determined whether these levels were due to redistribution and deposition of borate at a distance from the joint or if they were simply caused by the initial variation in borate content along the length. In the other two joints there was less borate found at the 140 mm position than found initially (49% in L-joint #3 and 21% in L-joint #11). In all five L-joints there was no borate found in the three sub-samples of the tenon itself.

The results indicate that there has been a substantial net loss of borate from L-joints in this field test and that loss has occurred through the break in the paint film at the joint. Given the excellent protection against decay that the boron diffusion treatment has afforded the L-joints for six years, this depletion was somewhat unexpected.

The L-joints in this study are in contact with plywood which remains wet for some time after rainfall events and this could have acted as a diffusion sink for the borate. The borate loss in this study was consistent with the results reported by Orsler (1993) for hyphen-joints with unpainted end-grain in a field test, and by Homan and Militz (1995) for painted blocks with a cut in the paint film in accelerated laboratory tests. However, in the first of these studies the experimental design did allow for water pooling around the test unit and in the later test the blocks were immersed in liquid water. Borate losses in accelerated laboratory tests have been found to be minimal when the paint film completely covered the wood and remained uncompromised (Homan and Militz 1995, Peylo and Willeitner 1995). None of these previous studies have evaluated the effect of leaching on durability of the borate-treated wood.

The losses of borate in this and other studies appear to contrast with the excellent performance of borate-treated wood under a three-coat paint finish in New Zealand (Drysdale 1994). However, it should be recognised that the L-joint represents a higher leaching and biodeterioration hazard than a window. Whereas a window receives some protection from the building and has a number of features designed to shed water, the L-joint has no protection and is designed to trap water. Furthermore the paint film of the L-joint is broken

immediately prior to installation whereas paint failure might not occur until some later time during the service life of a window.

The excellent performance of borate-treated L-joints in this accelerated test, despite considerable borate loss, may also be partially explained by the difference between the levels of preservative required to prevent mycelial growth and the lower levels required to prevent spore germination (Morton and French 1966). It is widely assumed that colonisation of wood exposed above ground occurs through germination of basidiospores. It is possible that diffusion of borate towards the joint from the distal end of the tenon has kept borate levels sufficiently high to prevent germination. Whether this protection will continue, given the low residual levels of borate, remains to be seen.

Since no analysis data are available on the other preservatives in test, no conclusions can be drawn as to whether depletion had occurred with these active ingredients.

4.0 Conclusions

Results from five to six years of L-joint testing suggest that a range of formulations could be effective replacements for pentachlorophenol in Canadian millwork.

5.0 References

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TABLE 1
Chemical retentions by uptake in Buckman tenon units

Treatment	Active Ingredient % m/m	Solution Retention kg/m ³				a.i. Retention kg/m ³			
		Dip		Double-vac		Dip		Double-vac	
		Mean	SD*	Mean	SD	Mean	SD	Mean	SD
TCMTB	0.15	15.0	(1.4)	32.2	(6.4)	0.023	(0.002)	0.048	(0.010)
	0.20	14.5	(2.0)	30.1	(4.5)	0.029	(0.004)	0.060	(0.009)
	0.50	12.8	(3.0)	25.4	(5.4)	0.064	(0.015)	0.132	(0.027)
TCMTB /IPBC (50:50)	1.00	13.3	(2.1)	23.5	(6.9)	0.133	(0.021)	0.235	(0.069)
	0.15	14.8	(2.2)	30.0	(4.6)	0.022	(0.003)	0.045	(0.007)
	0.20	12.9	(1.4)	23.8	(7.1)	0.026	(0.003)	0.048	(0.014)
	0.50	11.2	(2.1)	17.8	(5.4)	0.056	(0.011)	0.089	(0.027)
	1.00	10.8	(1.2)	21.9	(4.1)	0.108	(0.012)	0.219	(0.041)

* standard deviation

TABLE 2
Chemical retentions by uptake in Protim Solignum tenon units

Treatment	Active Ingredient % m/m	Solution Retention kg/m ³						a.i. Retention kg/m ³					
		Dip		Double-vac		Dip		Double-vac		Dip		Double-vac	
		Mean	SD*	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
TBTO	1.0	10.3	(2.0)*	24.2	(4.2)	0.103	(0.020)	0.242	(0.042)				
Propiconazole	1.0	ND		36.4	(5.8)	ND		0.364	(0.058)				
Zincneodecanoate	3.0	ND		26.5	(6.4)	ND		0.794	(0.191)				

* Mean (standard deviation)

ND - Not done

TABLE 3
Chemical retentions by uptake in reference material

Treatment	Active Ingredient % m/m	Solution Retention kg/m ³				a.i. Retention kg/m ³			
		Dip		Double-vac		Dip		Double-vac	
		Mean	SD*	Mean	SD	Mean	SD	Mean	SD
PCP	5.00	6.6	(0.9)	31.0	(5.4)	0.328	(0.041)	1.597	(0.315)
Mineral spirits		19.6	(2.8)	49.3	(8.3)				

TABLE 4

Boric acid content initially and after five years exposure

L-Joint	Initial %BAE Content	%BAE After 5 Years	5 year/Initial (%)
3	0.49 (0.17)**	0.08 (0.10)	16
11	0.19 (0.07)	0.04 (0.08)	21
23	0.28 (0.05)	0.09 (0.13)	32
26	0.23 (0.07)	0.09 (0.14)	39
32	0.20 (0.09)	0.06 (0.09)	30

* initial %BAE was a mean of nine sub-samples taken at one location; %BAE at 5 years was a mean of 75 sub-samples taken at nine locations

** the standard deviation is given in parentheses

TABLE 5

Decay ratings of Buckman L-joints after three and five years exposure

Treatment		Decay Ratings				
		3-yr		5-yr		
		Mean	SD	Mean	SD	
Mineral spirits	dip	1.6	1.3	2.0	1.4	
	double-vac	0.1	0.4	0.8	1.1	
TCMTB	dip	0.15%	0.2	0.6	0.6	1.1
		0.20%	0.0	0.0	0.2	0.6
		0.50%	0.2	0.6	0.7	1.1
		1.00%	0.0	0.0	0.0	0.0
	double-vac	0.15%	0.0	0.0	0.1	0.4
		0.20%	0.1	0.2	0.2	0.6
		0.50%	0.0	0.0	0.0	0.0
		1.00%	0.0	0.0	0.0	0.0
TCMTB & IPBC (50:50)	dip	0.15%	0.1	0.2	0.5	0.9
		0.20%	0.1	0.4	0.8	1.1
		0.50%	0.0	0.0	0.7	1.0
		1.00%	0.0	0.0	0.0	0.0
	vac	0.15%	0.0	0.0	0.1	0.4
		0.20%	0.1	0.4	0.2	0.6
		0.50%	0.0	0.0	0.1	0.3
		1.00%	0.0	0.0	0.0	0.0

TABLE 6**Decay ratings of Protim Solignum L-joints after three and six years exposure**

Treatment	3 years		6 years	
	Mean	SD*	Mean	SD
TBTO - Dip	0.00	0.0	0.2	0.6
TBTO - Double-vac	0.00	0.0	0.2	0.6
Propiconazole - Double-vac	0.00	0.0	0.00	0.0
Zinc neodecanoate - Double-vac	0.00	0.0	0.00	0.0

* standard deviation

TABLE 7**Decay ratings of reference material after five and seven years exposure**

Treatment	5 years		7 years	
	Mean	SD*	Mean	SD
5% PCP - Dip	0.3	0.8	0.6	1.1
5% PCP - Double-vac	0.00	0.0	0.00	0.0
Mineral spirits - Dip	2.5	1.4	3.0	1.2
Mineral spirits - Double-vac	1.8	1.2	2.7	1.2

* standard deviation

TABLE 8
Decay ratings of borate-treated material after 0, 1, 2, 3, 4, 5, and 6 years exposure

	0 year		1 year		2 years		3 years		4 years		5 years		6 years	
	Mean	SD*	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Untreated L-joints	0.0	0.0	NA	NA	0.6	0.8	1.2	1.3	2.0	1.7	2.3	1.6	2.7	1.5
Borate-treated L-joints**	0.0	0.0	NA	NA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

* standard deviation

** 5 borate-treated L-joints were sacrificed for analysis in 1995

NA data not available

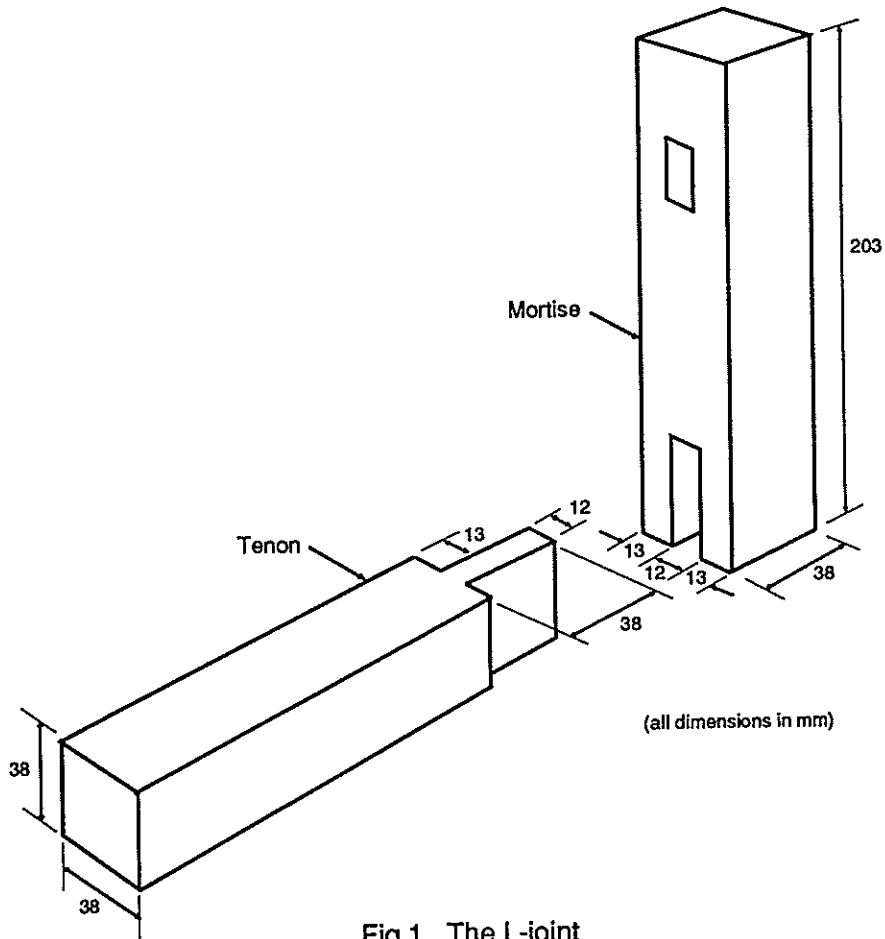


Fig.1 The L-joint

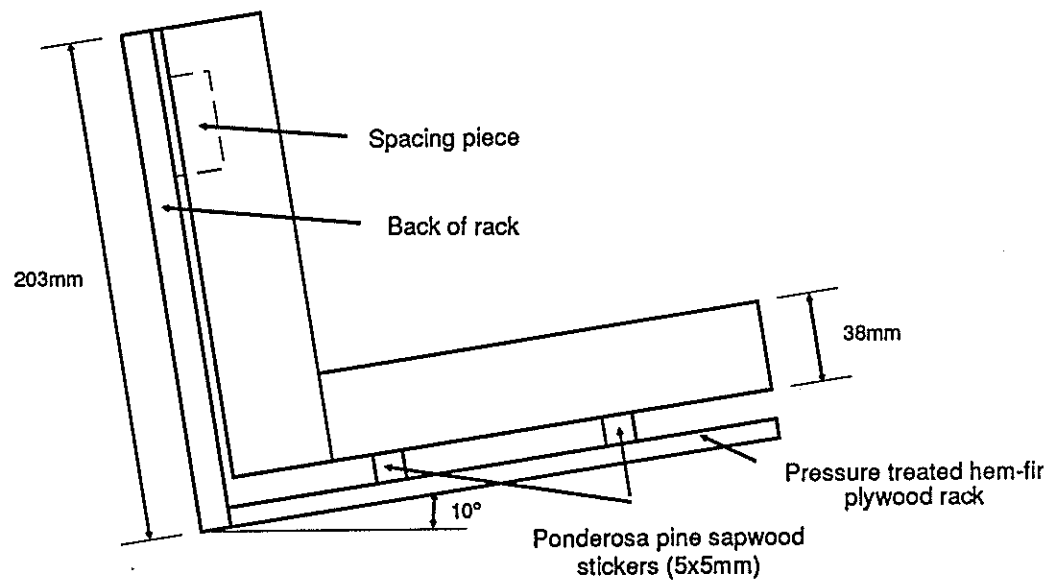


Fig.2 L-joint during exposure

FIGURE 3: BORATE CONTENT AFTER 5 YEARS EXPOSURE
IN CROSS-SECTIONS TAKEN ALONG TENON #3

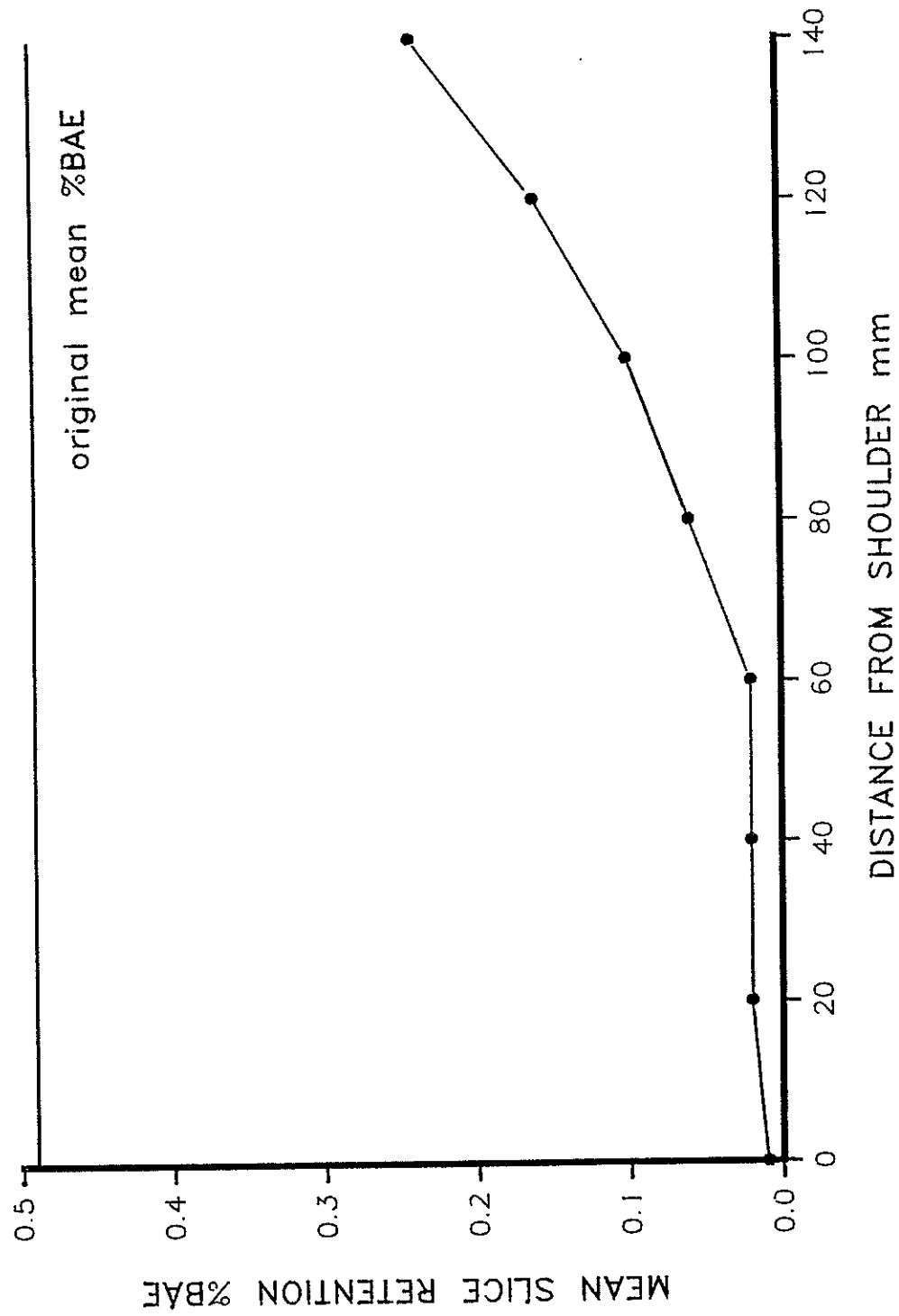


FIGURE 4: BORATE CONTENT AFTER FIVE YEARS EXPOSURE
IN CROSS-SECTIONS TAKEN ALONG TENON #11

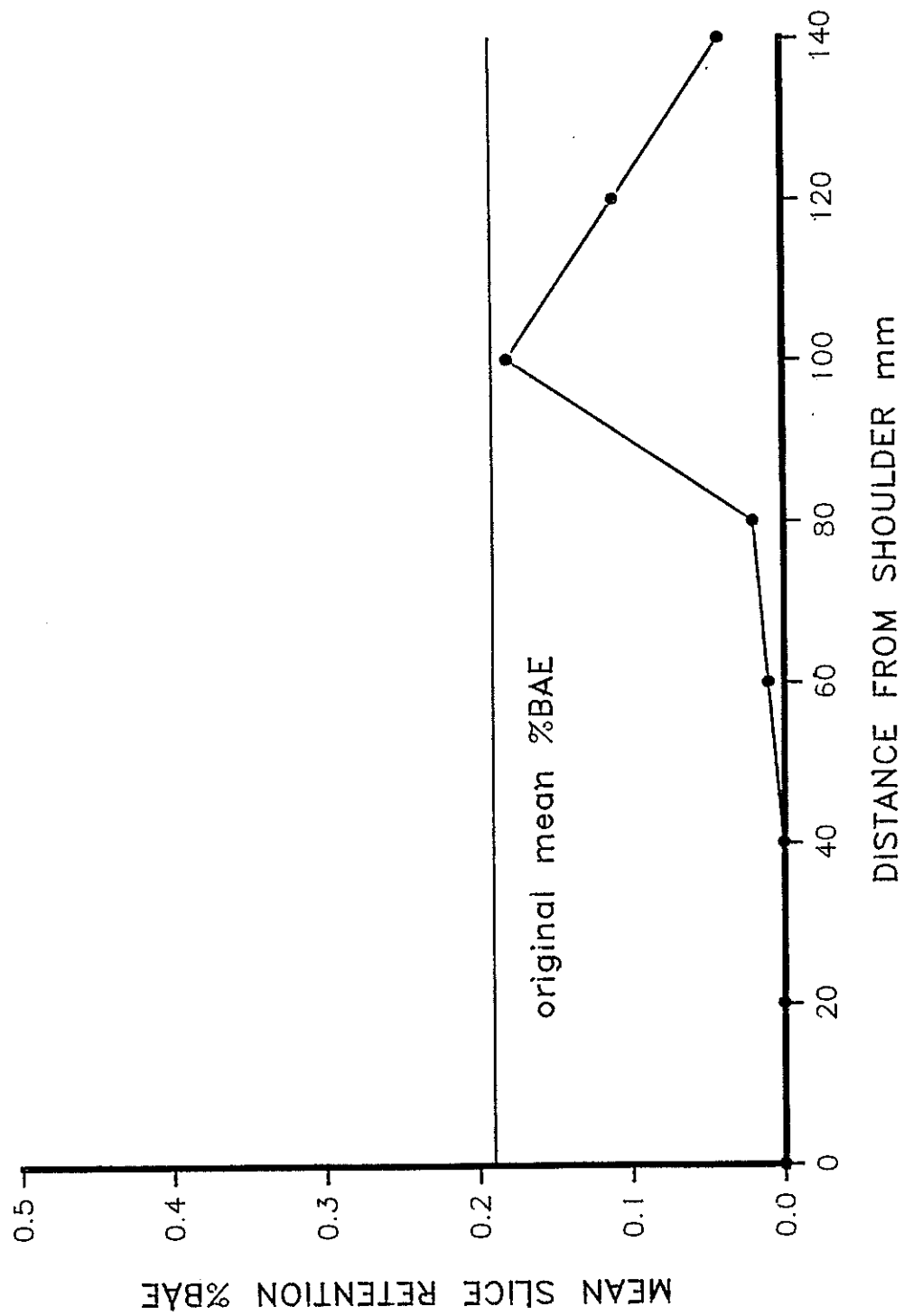


FIGURE 5: BORATE CONTENT AFTER FIVE YEARS EXPOSURE
IN CROSS-SECTIONS TAKEN ALONG TENON #23

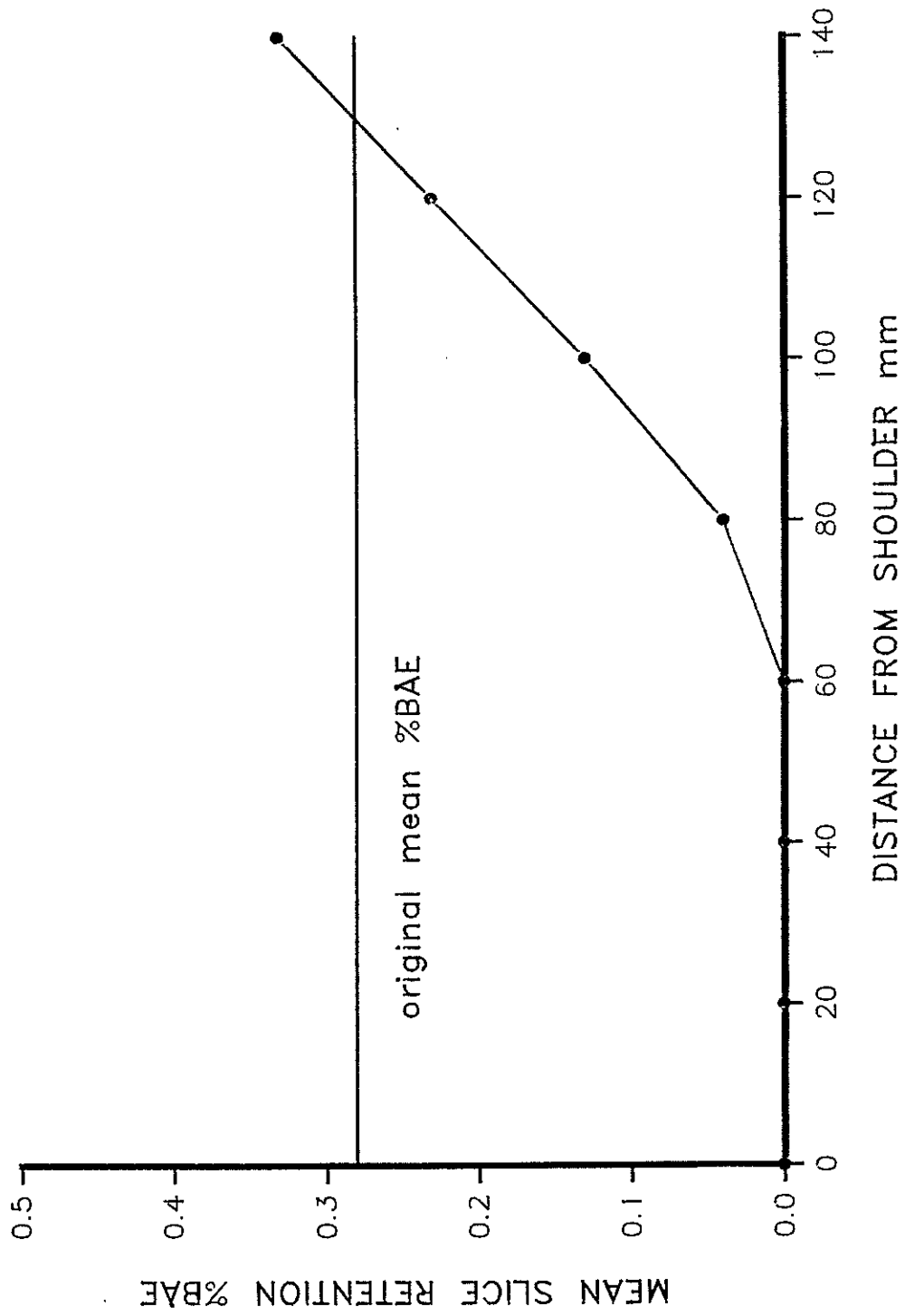


FIGURE 6: BORATE CONTENT AFTER FIVE YEARS EXPOSURE
IN CROSS-SECTIONS TAKEN ALONG TENON #26

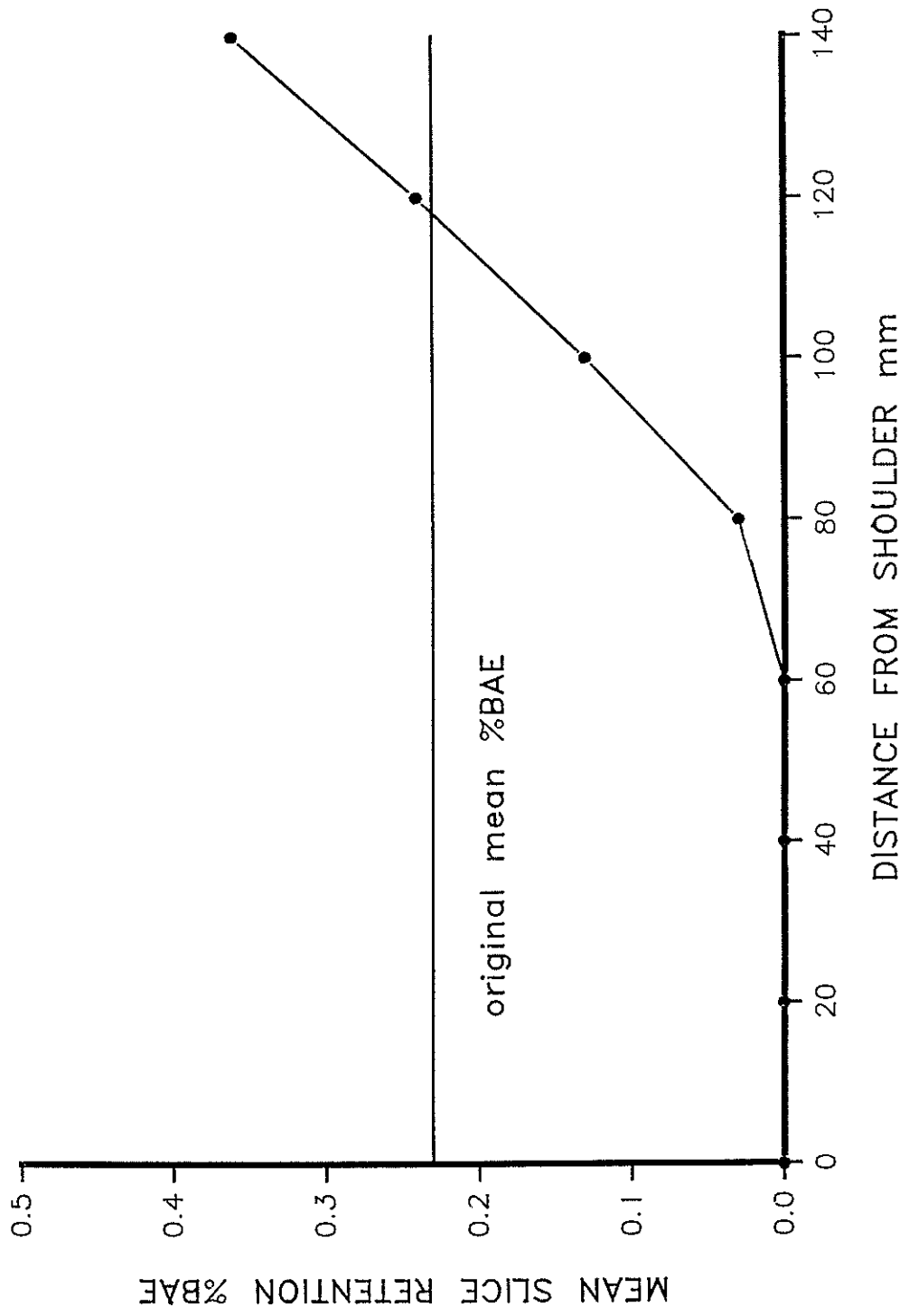


FIGURE 7: BORATE CONTENT AFTER FIVE YEARS EXPOSURE
IN CROSS-SECTIONS TAKEN ALONG TENON #32

