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EFFECTS OF DECOMPOSITION PRODUCTS OF PVC IN FIRE ON STRUCTURAL CONCRETE

by

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SUMMARY

Full scale fire tests have been conducted in buildings to compare the effect of combustion products on concrete building elements when the fire load was totally cellulosic and when 30 per cent of the fire load was PVC. After the fire the buildings were kept under observation and at intervals concrete roof elements were removed and loaded to structural failure. Samples of the concrete were then analysed for chloride content.

The tests have shown that in fires involving PVC, chloride deposition can occur on concrete surfaces under both dry and humid conditions. Observations and analyses of the concrete for periods of up to 13 months after the fires showed no indications that the building suffered structurally because of the effects of the chloride. Under the conditions of these tests, corrosion is unlikely to be a problem in dense concrete constructions whether of a reinforced or prestressed nature provided the relevant British Standard Codes of Practice have been complied with.

KEYWORDS: Corrosion, PVC fires, structural concrete.

DEPARTMENT OF THE ENVIRONMENT AND FIRE OFFICES' COMMITTEE
JOINT FIRE RESEARCH ORGANIZATION

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INTRODUCTION

The April 1968 edition of Schaden Spiegel[1] contained a report under the title 'Burning of plastics and its consequences'. The document concentrated on PVC and the corrosive effects of the hydrogen chloride released when this material is burnt. Several case studies occurring in the period 1959-68 were cited and often a substantial portion of the damage was attributed specifically to the burning of plastics. This report brought to a focus the growing concern over the problem of corrosion subsequent to the involvement of PVC in fires.

Towards the end of 1968 a meeting was held at Boreham Wood attended by representatives from: the Plastics Division of Imperial Chemical Industries (ICI), Rubber and Plastics Research Association (RAPRA), the Fire Offices' Committee (FOC) and the Joint Fire Research Organisation (JFRO). Many aspects of the corrosion problem were discussed and it was agreed that whilst numerous incidents had been reported, it was difficult to be specific about the hazard or the solution as little quantitative data existed. It was proposed that experiments should be carried out under controlled conditions to provide the data enabling a proper evaluation of the extent of the problem.

In the case of a fire, both the building and its contents are placed at risk. Of particular concern was the possibility of corrosion damage to the fabric of building structure which had otherwise maintained its stability. It had been alleged that prestressed and reinforced concrete structures were particularly susceptible to corrosion damage as the chloride ions would migrate into the concrete and cause corrosion of the steel. By its nature this would be a long term process which may not evidence itself for several years. The decision to write off or reinstate a building after a fire invariably has to be made fairly quickly. It is also important from the point of view of safety that one should be able to make a valid assessment of the structural state of a building following a fire.

Following the discussion a programme of work was planned which would examine the effect on concrete structures of being exposed to a fire involving the combustion of PVC with resultant exposure to hydrogen chloride (HCl). It was felt that this was an aspect of the problem which needed immediate attention.

It is the purpose of this note to describe some full scale tests that were carried out at the Fire Research Station over the period 1969-72. The work forms part of a co-operative research programme undertaken by the Joint Fire Research Organisation and ICI.

Since the commencement of the programme there have been many articles published dealing with related problems and the main references are appended at the end of the report[2-9]. As far as fires within the United Kingdom are concerned, a search was made for any instances cited where there was evidence of corrosion resulting from the combustion of PVC. Only two published case histories were found, relating to fires at a shoe factory[10], and at the chemical laboratories of Bristol University[11,12].

2. THE TEST PROGRAMME

Initially, the proposals were that the work should be carried out in two distinct phases. Experiments on a laboratory scale would give basic data on hydrogen chloride decomposition and rate of chloride penetration into concrete. This work would establish the parameters for large scale ad hoc tests carried out to confirm the performance of full size structural elements.

2.1. Small scale laboratory tests

These tests were to be carried out by ICI and RAPRA to establish a suitable analytical method for the determination of chloride in concrete, a systematic examination of the variables affecting chloride deposition, its effect on the compressive strength of concrete, and its effect on the bond between concrete and steel reinforcement.

Only a part of this work was completed before proceeding with the full scale tests. The analytical method for chloride determination is described in Appendix 1.

2.2. The full scale ad hoc tests

A comparative test was designed in which PVC comprised about 30 per cent of the fire load, the balance consisting of cellulosic material. This simulated a mixed storage situation and represented a generally agreed compromise between the extremes of proportions of PVC likely to be met in practice. It was envisaged that such a proportion could occur, for example, in a warehouse containing goods fabricated from

PVC together with other combustible items. A control test involving only cellulosic materials provided a reference point for determining effects solely due to the fire.

The test building was in two separate halves, each having three compartments. The tests were designed so that the hot gases from a centrally positioned fire chamber could flow outwards through two adjacent rooms, in one of which the humidity was increased by a water spray. There were therefore six defined situations:

Test (A)
- Control fire - fire compartment
- Control fire - dry side chamber
- Control fire - humid side chamber

Test (B)
- PVC fire - fire compartment
- PVC fire - dry side chamber
- PVC fire - humid side chamber

The division of responsibilities in the tests was as follows:

ICI - The provision of PVC and the analysis of fire gases and the sampling and analysis on concrete specimens from the tests. (The analysis of the concrete was carried out by RAPRA on behalf of ICI).

JFRO - Design and running of tests and determination of the structural performance of elements.

Tests A and B were carried out on 16 and 18 June 1970 respectively. A sampling technique was adopted for the fire gases; the samples obtained were later analysed in the laboratory. Data obtained from the gas samples indicated that the hydrogen chloride levels were extremely low. Subsequent investigation showed that absorption may have taken place in the sampling lines and the test involving PVC fire load was therefore repeated with improved sampling techniques. This repeat test (Test C) was carried out on 24 August 1971 after reinstatement of the test building.

3. THE EXPERIMENTAL DESIGN FOR THE AD HOC TESTS

3.1. The structural elements

The structural elements under test were hollow prestressed concrete floor units supplied by Concrete Ltd. The units were 133 mm deep, each having 8 No.10 SWG prestressing wires. The 28-day strength of the concrete was stated to be 41 N/mm^2

(6000 lb/in^2). A typical unit is illustrated in Fig.1. A superimposed load of 413 kN/m^2 (60 lb/ft^2) was applied to the units using bricks as deadweight (Plate 1). The load remained on the units until they were removed for structural tests and subsequent analysis.

3.2. The test building

The test building, approximately 9 m x 4.6 m (30 ft x 15 ft) in plan, was constructed from aerated concrete blocks on a concrete plinth and was divided into two halves so that the PVC and control fires could be conducted separately. The units under test were supported on a steel frame and formed the roof of the test building. Concrete block infill walls protected the steel frame from the effects of the fire.

Each half of the building was sub-divided into three compartments as shown in Fig.2; doors and ventilation openings were provided as indicated. A general view of the building is shown in Plate 2. Each compartment measured internally approximately 3.96 m x 1.2 m x 2.4 m high (13 ft x 4 ft x 8 ft high). The windows to the fire chambers each provided 1.4 m^2 (15 ft^2) of ventilation area when fully open. Doorways communicated with each side chamber, and high level openings 0.1 m^2 (1 ft^2) in area vented from these chambers to the cutside. All the openings were provided with adjustable shutters so that the flow of hot gases could be controlled. To encourage an equal amount of gases to flow out through the side chambers, the vents were regulated so that the temperature distributions in the humid and dry side chambers were comparable.

3.3. Fire loads

Control fire (Test A)	113 kg (250 lb) of 50 mm x 50 mm (2 in x 2 in) softwood in 0.76 m (2 ft 6 in) lengths. The timber was arranged in two cribs, with 1:1 spacing, symmetrically situated in the fire chamber. A small amount of fibre insulating board was used to aid ignition.
First PVC fire (Test B)	In this test the full timber fire load of the control test was augmented by an additional 53 kg (117 lb) of PVC. The PVC component was evenly distributed between the two cribs and consisted of: i) 44.8 kg (99 lb) of 25 mm (1 in) diameter solid PVC rod in 0.76 m (2 ft 6 in) lengths, inserted into the crib. ii) 8.2 kg (18 lb) of PVC gutter section laid on top of the cribs.

Repeat PVC fire (Test C)

In the first PVC fire the arrangement of the PVC part of the fire load had not been entirely satisfactory as the PVC incorporated in the crib restricted ventilation and slowed the rate of combustion. Before the repeat test was carried out, trials were conducted in which a more satisfactory arrangement was established. In the second test, therefore, the PVC was supplied in the form of 76 mm (3 in) profile corrugated sheet which was attached to the walls in a double layer to give a PVC fire load of 59 kg (130 lb) in addition to the 113 kg (250 lb) of timber fire load.

3.4. Humidity

Water was continuously introduced into the humid chambers over the test period using a pair of No.1 Bray jets which gave a finely atomised spray. The nominal input was at a rate of 90 l/h (20 gph)

3.5. Measurements

Temperatures in each of the chambers were monitored continuously in each of the tests. Three complete concrete units supported at the ends spanned each compartment and the central vertical deflections of the units were recorded at intervals before and after the test. During the tests the deflection of the middle unit in each compartment was measured. Sampling of the gases (Plate 3) was undertaken by ICI and the samples were subsequently analysed at their Welwyn Garden Laboratory. The residual strength of the concrete units at different times after the test was determined by removing one unit from each compartment of the building and applying a uniformly distributed load on simple end supports. Load deflection characteristics were recorded for each unit, the load being increased until failure occurred. Samples from these units were then analysed by RAPRA for the determination of chloride ion penetration.

3.6. Additional samples

Prestressed concrete units were chosen because any chloride corrosion of the steel, if it occurred, would be more critical than in the case of reinforced concrete units. Reinforced concrete is however generally of a lower strength and could be expected to be more permeable to chloride diffusion. In order to examine the performance of reinforced sections a number of additional samples were included in the tests as below:

1. Reinforced concrete lintels manufactured at the Fire Research Station, nominal 1:2:4 mix with reinforcing bars having 12 mm, 25 mm and 36 mm nominal depth of cover. These were exposed in both tests B and C.

2. Concrete cubes and cylinders, some containing embedded steel bars.

3. Commercially manufactured reinforced concrete fence posts in test C only.

The reinforced concrete lintels were incorporated into the building spanning the intercommunicating doorways. In this position they were exposed to the full flow of gases passing through the fire chambers. The other samples were mounted on stands in the test chambers (Plates 4 and 5).

As an addition to the main experiment, a number of electrical relays were mounted close to the concrete cubes and cylinders (Tests A and B) to obtain some data on the possible corrosion of electrical equipment.

4. TEST RESULTS

4.1. Preliminary laboratory tests at RAPRA

This work is recorded under RAPRA Research Report No.9642 - June 1969[13]. Two tests were carried out in a cubic metre box, one side of which consisted of a sliding door which provided adjustable ventilation for the fire. One wall of the box was lined with PVC sheet, another with hardboard and the third with a concrete slab 50 mm (2 in) thick. A reinforced concrete beam was suspended above the fire and protruded from the front sliding door. A third concrete sample was suspended 0.6 m (2 ft) from the vent. The main fire-load consisted of 6.4 kg (14 lb) of fibreboard strips and 3 kg (6.6 lb) of PVC pipe.

The first fire was well ventilated and vigorous burning resulted in temperatures of 700°C. In the second fire ventilation was restricted and combustion was much slower, the temperatures only reaching 350°C at the vent. Concentration of hydrogen chloride inside the box was determined as 12,100 ppm in the vigorous fire and 6640 ppm in the fire with the restricted ventilation. When sections from the beams were analysed the highest chloride in concrete concentration recorded was 0.48 per cent at the surface of the beam from the more severe fire. At a depth of 42 mm below the surface the chloride in concrete level was 0.10 per cent. Analysis of a control sample (no exposure) gave corresponding figures of 0.003 per cent and 0.002 per cent. After the above beam had been subjected to a humidity cycling and wetting routine for 35 days, surface chloride concentrations had dropped to 0.10 per cent and the level at 42 mm depth had increased to 0.14 per cent.

4.2. Full scale ad hoc tests

4.2.1. Air temperatures - Tests A and B

Data from the temperatures recorded during the tests are given in Figs 3, 4 and 5. The fire in the control test (Test A) was allowed to burn for a 30 minute period and maximum temperatures approaching 650°C were achieved in the fire chamber. In the first PVC fire that followed (Test B), the position of the PVC components in the cribs significantly reduced the rate of growth of the fire. For this reason, the test was continued for a 60 minute period and even then only 37 kg of PVC was burnt. In this test the temperatures were much lower, generally being maintained around 450°C. In each test the temperatures in the dry and humid chambers were comparable with each other but, as in the case of fire chamber temperatures, the values recorded in the control fire were higher.

4.2.2. Other measurements (Tests A and B)

The maximum concentration of moisture vapour was 17.8 per cent in the humid compartment during the control test. The scatter on these results was however very large, and a figure of 15 per cent was recorded in one of the dry compartments. No concentration of chloride greater than 10 ppm was recorded in any of the gases sampled from either test.

Observers at the tests were agreed that subjectively (on the basis of pungency) there were fairly high concentrations of hydrogen chloride in the gases coming off from the PVC fire. Also taking into account the quantity of PVC consumed it is not feasible that all of the hydrogen chloride produced would have been lost by through ventilation across the building.

4.2.3. Air temperature - Test C

Temperature records for the repeat PVC test (Test C) are also included in Figs 3, 4 and 5. The fire was appreciably hotter than the control test and was allowed to burn for 47 minutes. About 50 kg of PVC were consumed. The temperatures measured in the side chambers were comparable to those obtained in the control test (Test A).

4.2.4. Hydrogen chloride levels - Test C

The results of the chemical analysis are given in Fig.6. In this test hydrogen chloride levels in excess of 10,000 ppm were recorded. These agree fairly well with the values recorded in the laboratory tests at RAPRA.

4.2.5. Post-test visual inspections - prestressed units

Inspection of the compartments after the fires showed them to be generally free from major structural damage. An exception were the roof units in the fire compartment of the second PVC fire (Test C) which exhibited longitudinal cracking along the soffit. The centre unit was particularly badly damaged and this showed up in the residual strength tests when it collapsed under a load of 182 kg whereas the other units supported loads in the region of 2,500 kg. The appearance of the inside of the building after the tests is shown in Plate 6. There were fairly heavy soot deposits in the side chambers in all the tests and although no quantitative measurements were made these appeared to be significantly greater in the PVC fires. The temperatures in the fire chambers were too high to allow extensive soot deposition.

4.2.6. Structural tests on prestressed units

Each compartment was spanned by three units (Plate 7). In the case of the first two tests (A and B) a single unit was removed from each compartment at 7 days, 3 months, and 10 months after the test. Units from the repeat PVC fire test (Test C) were removed at 1, 6 and 13 months. The differences in the removal times of Tests A, B and C is fortuitous and has no significance.

After removal, each unit was simply supported at its ends and a uniformly distributed load consisting of cast iron weights was applied until failure occurred (Plate 8). The loads sustained by each unit at failure and the deflections produced are given in Table 1. A histogram of the failure loads is given in Fig.7 and typical load-deflection curves are shown (for Test C) in Fig.8.

Deflections of the units before and after the test but while still on the building were recorded and Fig.9 gives results from Test C for units remaining in situ for 13 months. In all three tests, the units did not deflect in the vertical direction to any significant extent before or after the fires. While they were being heated during the fires however, there was some downward deflection. In Test A, units over the fire chamber deflected downwards to a maximum of 21 mm but returned to their original position after the test. In Test B, where lower temperatures existed, a downward deflection of only 13 mm was observed and again the units returned to their original position. The maximum deflection during Test C was 63 mm and the units did not fully recover - the residual deflection at 13 months being 10 mm. The hotter fire in this test led to permanent deformation to the units (Fig.9) possibly because of some relaxation of pre-stress. As mentioned in 4.2.5 the fire chamber units suffered damage.

4.2.7. Chemical analysis - prestressed units

Samples of concrete from each unit were sent to RAPRA for analysis. The initial analysis was carried out on receipt of the samples, additional material being stored at RAPRA under both dry and humid conditions for periods of up to 6 months. The results of the analysis on concrete samples are the subject of RAPRA Consultative Reports Nos D 9566, D 9618, D 9675, D 9735, D 10084, D 10573 and CTR 33. The analysis techniques are given in Appendices I-III summarised results being illustrated in Figs 10 and 11 and Tables 2-4.

Figures for chloride content quoted in this report with the exception of those in para 4.1 are on the basis of equivalent anhydrous $CaCl_2$ percentage in cement. These were determined following an analysis of the concrete as reported in the Appendix.

In addition to the chemical analysis, the condition of the prestressing wires in the units was assessed visually immediately after the loading tests. The Cement and Concrete Association were invited to examine samples from the above units including one where some rusting of the prestressing wires had also stained the concrete. They reported that the degree of corrosion evident on the steel was not at variance with that which would be expected under normal exposure conditions. Colorimetric indicator tests in which phenolphthalein was applied to the freshly broken concrete, were also carried out on the slabs from the second PVC fire (Test C) which were loaded at thirteen months. A pink colour developed within the concrete indicating a predominately alkaline condition but there was however an unstained edge indicating an acidic condition. Whilst this was in part due to chloride contamination it could also be due to carbonation which would take place during normal atmospheric exposure. (Plate 9 illustrates this test applied to a concrete test cube).

4.2.8. Other concrete samples

The results of crushing small concrete samples and the approximate range of temperatures to which these were exposed are shown in Table 5. The cylinders, 150 mm dia, had 12 mm diameter mild steel bars along the central axis and when split open by the indirect tensile test method, showed some corrosion along the bar to a depth of 12 mm from the exposed length. The cubes with 12 mm steel bars through their centres were originally intended for bond strength determination after exposure. When fractured, the steel was found in a bright condition indicating hardly any loss in the bond strength. Some of these samples showed corrosion to a depth of 12 mm measured from the point where the steel broke the

surface. This was attributed partly to the development of cracks at the steel concrete interface caused by non-compatible rates of thermal expansion. Additional tests using ultra violet light and fluorescent penetrants did not however reveal any significant cracking.

Table 5 also shows the results of compressive tests on 100 mm cubes.

Table 5. Small concrete samples

Sample	Exposure	Environment			Exposure temperature
		Humid	Fire chamber	Dry	
		N/mm^2	N/mm^2	N/mm^2	°C
Cubes					
	Test A	39.1	36.2	39.1	100-300
(Compressive strength)	Tests B & C	46.7	46.0	39.7	100-250 and 200-300
Cylinders					
	Test A	1.35	1.35	1.62	100-300
(Indirect tensile strength)	Test B	2.03	1.53	1.67	100-250

4.2.9. Lintels

The lintels removed from the building had been exposed to the full severity of both fires involving PVC (Tests B and C), and during the first fire fine cracks had appeared in the concrete. Sections of these lintels were examined by the Cement and Concrete Association who reported that the degree of corrosion evident on the steel was consistent with that which would be expected under normal exposure conditions.

4.2.10. Fence posts

Reinforced concrete fence posts included in the second PVC fire (Test C) were particularly interesting as analysis of control samples showed a very low inherent chloride content (0.01%) and analysis of the exposed specimens indicated a fairly high surface concentration after the fire, 1.4 per cent in the dry chamber and 1.95 per cent in the humid chamber. Further tests after 12 months storage under normal laboratory conditions however, showed that the chloride content was only 0.1 per cent at a depth of about 10 mm. This data is given in Table 6 and shown in Figure 12.

4.2.11. Electrical relays

The electrical relays included in the tests all suffered temperature damage, and as a consequence no check could be made on their operating parameters. After

the fires a visual examination revealed smoke and heat damage but no obvious signs of corrosion. At this stage half of the relays were carefully washed and dried using a proprietary solution used for removing smoke deposition. These together with the uncleaned relays and some control samples were again divided and stored under humid and dry conditions for a period of six months. In all cases the untreated exposed samples corroded badly and Plate 10 illustrates the difference between washed and unwashed samples after 6 months humid storage.

5. DISCUSSION

The aim of the tests was to obtain an indication of the effect on concrete structures of being exposed to a fire involving the combustion of PVC and to the resultant hydrogen chloride in the fire gases. This was achieved by comparing the performance of structural elements subjected to a control fire involving only cellulosic materials, with identical elements exposed to fire conditions in which 30 per cent of the fire load was PVC. The tests were designed so that combustion products from the fires were induced to flow through adjacent chambers, referred to as the 'dry' and 'humid' chambers. The fires themselves were designed so that the elements in the side chambers although exposed to high temperatures, would not suffer structural damage because of the fire. An attempt was made to control the fires and flow of combustion products, so that the temperatures in the humid and dry compartments were similar, and also so that comparable temperature regimes prevailed in the cellulosic fire (Test A), and the fires incorporating PVC (Tests B and C).

The cellulosic fire (Test A) was conducted first, and average temperatures in the fire compartment just exceeded 600°C at ceiling level. In the side compartments the temperatures reached a little over 400°C and reference to Figures 4 and 5 shows that the temperatures in the dry and humid compartments were very similar.

The first fire involving PVC (Test B) did not result in temperatures comparable with those in the control fire. This was caused by the arrangement of the fuel so that soon after ignition, softened PVC fused together and restricted ventilation to the cellulosic content of the fire load. Except for an initial peak the temperatures were about 150-200° lower than those of the control fire. Even the extension to 60 minutes resulted in only 70% of the PVC fire load being consumed.

When the results of the chemical analyses became available it was evident that the sampling of hydrogen chloride had not been satisfactory and eventually it was concluded that this was probably because of absorption of the hydrogen chloride by the heated stainless steel sampling lines. As a consequence ICI developed an improved sampling method for the repeat test. Because the development of the PVC

fire had also not been satisfactory, exploratory tests were carried out to establish a more suitable fuel arrangement. The temperatures in the repeat PVC fire (Test C) Figs 4 and 5, were much closer to those of the control fire in the dry and humid compartments. Figure 3 compares the temperatures in the fire compartments for the three fires. The second PVC fire was hotter than the control fire in the final stages because the total combustible content was greater.

After the fires visual observation of the building revealed no obvious deterioration. Deflection measurements made on the units at regular intervals indicated no further deterioration in structural strength. The general level of variation in strengths of units (Fig.7) is within the limits expected with the type of unit and the loading methods used. The three units from the fire chamber in the second PVC fire suffered heat damage and gave lower strengths. Three additional control units were loaded hydraulically and gave a mean failure load of 3070 kg (6750 lb) (Plate 11).

The results of the chloride analyses are summarised in Figures 10 and 11. Figure 10 shows that the percentage of chloride and the range of values measured in the surface layer of the concrete exhibited an overall tendency to decrease with time. This is consistent with some diffusion of the chloride ions into the concrete and possibly some loss from the surface. Figure 11 shows that the chloride levels on the interior surface of the concrete did not vary significantly with time over the duration of storage examined. At the depth where the prestressing wires were located, analysis shows an equivalent percentage anhydrous calcium chloride greater than zero without any significant difference between units exposed to the different types of fire.

The concrete fencing posts were examined in more detail and reference to Figure 12 shows no evidence that much diffusion had occurred. A chloride concentration of 0.10 per cent at one month at a depth of 10 to 25 mm had not increased after a further 11 months storage.

CP 110 : Part 1 : 1972 paragraph 6.2.4.2 recommends that the level of anhydrous calcium chloride in the cement should be zero for prestressed concrete and should not exceed 1.5 per cent by weight of cement for reinforced concrete. The level of calcium chloride on the exposed surface of the units was found to be between 0.5–1 per cent.

6. CONCLUSIONS

Full-scale ad hoc fire tests have been performed to determine the effect on concrete elements, mainly prestressed concrete slabs, of being exposed to a fire involving the combustion of PVC and to the resultant hydrogen chloride in the fire

gases. The following conclusions are drawn from this limited investigation conducted under the conditions described earlier:

1. The prestressed concrete units showed no significant reduction in strength when exposed to fires lasting up to 60 minutes in which the maximum air (ambient) temperature did not exceed 600°C.

2. When the fire temperature exceeded 800°C, the units showed a reduction in flexural strength of the order of 20 per cent immediately after the fire and no subsequent further reduction after a year.

3. None of the units exposed to temperatures between 200 and 600°C with the hydrogen chloride concentration during the course of the fire approaching 12,000 ppm have shown a significant reduction in strength either immediately after the fire or after a lapse of 13 months.

4. Visual examination of units 13 months after the fire test using phenol-phtlalein indicator techniques has not shown a penetration of chloride to depths greater than 2 mm.

5. Chemical analysis of the concrete from the units exposed to PVC fires have shown a surface concentration of chloride ions up to maximum of 2.3 per cent w/w expressed as $CaCl_2$ by weight of cement soon after a fire with a progressive decrease after 12 months to a maximum concentration of 1.5% w/w of cement.

6. The concentration of chloride ions on the surface of units subjected to non PVC fires was not more than 0.2% w/w of cement.

7. The concentration of chloride ion in the vicinity of the reinforcement was less than 0.4 per cent w/w of cement and did not increase after 12 months storage.

8. The tests did not indicate that the chloride ions had penetrated significantly deeper into the lower strength concrete used in fence post construction. This may have been due to the high cement content in the posts as determined by subsequent analysis (Appendix 1).

9. Where steel bars projected from the surface of concrete, corrosion of steel under the surface occurred but the damage did not extend beyond 10 mm.

10. Exposed electrical equipment such as relays is likely to suffer serious corrosion damage by the deposition of hydrochloric acid on the exposed surfaces. However, the amount of damage can be minimized by washing the surfaces with a mild detergent solution immediately after the fire.

7. ACKNOWLEDGEMENTS

These are due to Rubber and Plastics Research Association of Great Britain who on behalf of ICI conducted the analysis of the concrete samples in this programme and also to the Cement and Concrete Association who took part in the analysis procedure in the later stages.

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Table 1. Roof Slab Failure Loads

Delivery	Beams	Compartment	Removed from building	Failure load kg	Deflection at failure mm
1970	Control	Not fire tested	-	1910*	93
	Test A Cellulosic fire	Fire	7 days 3 months 10 months	2550 2680 2400	129 82 80
		Humid	7 days 3 months 10 months	2500 2770 2500	98 103 76
		Dry	7 days 3 months 10 months	2730 2860 2340	92 90 89
	Test B 1st PVC fire	Fire	7 days 3 months 10 months	2545 2590 2050	115 99 50
		Humid	7 days 3 months 10 months	2400 2180** 2360	68 55 98
		Dry	7 days 3 months 10 months	2520 2820 2450	97 83 103
1971	Control	Not fire tested	1 month 13 months	3040 2768 3000/ 3100/ 3120/	72 78 130 110 110
	Test C 2nd PVC fire	Fire	1 month 6 months 13 months	182+ 1950 1950⁎	13 84 64
		Humid	1 month 6 months 13 months	2950 2270 2910	84 105 85
		Dry	1 month 6 months 13 months	2860 2545 3140	84 66 87

* Damaged during structural test
** Damaged during removal from structure
/ Tested in a hydraulic loading machine
\+ Damaged by fire before structural tests
⁎ Damaged by fire but also badly made

TABLE 2. Test A Cellulosic Fire. Chloride Concentration - Roof Slabs

RAPRA Report No.	9566		9618		9618		9735		9735		9675	
Storage Time at RAPRA	Initial		3 months		3 months		6 months		6 months		3 months (at JFRO)	
Storage condition at RAPRA	On arrival at RAPRA		Dry		Humid		Dry		Humid		Outdoors (at JFRO)	
Sample Location	Int	Ext	Int	Ext	Int	Ext	Int	Ext	Int	Ext	Int	Ext
Control Spec.	0.15	0.05	-	-	0.24	0.16	0.16	0.03	0.12	0.05	-	-
Fire chamber	0.18	0.14	0.33	0.15	0.22	0.15	0.14	0.13	0.16	0.14	0.22	0.15
Dry chamber	0.11	0.19	0.02	0.02	0.03	0.02	0.06	0.02	0.04	0.02	0.03	0.01
Humid chamber	0.02	0.13	0.20	0.16	0.13	0.11	0.22	0.13	0.20	0.14	0.37	0.10

TABLE 3. Test B. PVC Fire. Chloride Concentrations - Roof Slabs

RAPRA Report No.	9566		9618		9618		9735		9735		9675	
Storage Time at RAPRA	Initial		3 months		3 months		6 months		6 months		3 months (at JFRO)	
Storage condition at RAPRA	On arrival at RAPRA		Dry		Humid		Dry		Humid		Outdoors (at JFRO)	
Sample Location	Int	Ext	Int	Ext	Int	Ext	Int	Ext	Int	Ext	Int	Ext
Control Spec.	0.15	0.05	-	-	0.24	0.16	0.16	0.03	0.12	0.05	-	-
Fire chamber	0.20	0.99	0.03	0.98	0.01	0.55	0.09	0.37	0.07	0.43	0.44	0.67
Dry chamber	0.02	1.95	0.05	1.55	0.09	0.83	0.19	0.43	0.05	0.57	0.02	0.79
Humid chamber	0.20	2.35	0.16	2.20	0.11	0.65	0.39	0.89	0.18	0.74	0.03	0.77

TABLE 4. Test C. PVC Fire. Chloride Concentrations - Roof Slabs

RAPRA Report No	10084			10244		10573			10573		
Storage time at JFRO	1 Month			6 months		13 months			12 months (at RAPRA)		
Storage condition at JFRO	outdoors			outdoors		Outdoors			Humid (at RAPRA)		
Sample Location	Int	Centre	Ext	Int	Ext	Int	Centre	Ext	Int	Centre	Ext
Control spec.	0.01	-	0.00	0.01	0.01	-	-	-	-	-	-
Fire Chamber	0.44	0.47	1.90	0.02	1.05	0.08	0.65	0.83	0.08	0.31	1.45
Dry Chamber	0.05	0.04	0.68	0.08	0.95	0.08	-	0.31	0.13	-	1.20
Humid Chamber	0.02	-	0.65	0.05	0.69	0.16	-	0.39	0.02	-	1.05

TABLE 6 Test C. PVC Fire. Concrete Fence Posts - Chloride Concentrations

RAPRA Report No	10084			No 33 (1973)					
Storage Time at JFRO	1 month			12 months					
Storage condition at JFRO	Laboratory			Laboratory					
Depth below surface (mm) / Sample Locn.	Ext	25	50	2.5	10	17.8	25	33	43
Control spec.	0.01	0.00	0.01	-	-	-	-	-	-
Dry chamber	1.40	0.01	0.03	-	-	-	-	-	-
Humid chamber	1.95	0.07	0.01	1.75	0.11	0.11	0.10	0.01	0.01

APPENDIX 1

CHEMICAL ANALYSIS BY RAPRA

Expression of chloride contents as equivalent anhydrous calcium chloride in cement

The main analyses of the concrete units, as described in Appendix II, determined the percentage concentration of chloride in the concrete by weight. It was however decided that it would be better to express the results as percentage equivalent anhydrous calcium chloride in cement. This was done mainly because it is the established practice in publications such as Codes of Practice for concrete to express chloride content in this way. It would also be expected that the chloride would be present predominately in the cement paste and not in the aggregate content of the cement.

All results for chloride content have therefore been expressed as equivalent anhydrous calcium chloride in cement and these figures have been obtained by an arithmetical conversion from the determined figures of chloride in concrete with the assumptions made that the concrete in the prestressed units contained 20.0 per cent and in the fence posts 18.5 per cent cement. The steps taken to justify the assumption on cement content were:

1. Duplicate cement samples from both units and fence posts were taken by RAPRA. One set was analysed by RAPRA for calcium and magnesium content in addition to the chloride content. The methods used to determine the calcium and magnesium contents are given in Appendix III.

   The other set of samples was subjected to a more detailed analysis by the Cement and Concrete Association (C & CA) for cement content using their standard techniques.

   From a comparison of these results it was concluded that a reasonably accurate determination of the cement content could be obtained using only the calcium content measured by RAPRA and converting this to cement assuming the cement contained 64.5 per cent calcium oxide (CaO). (It had been established by C & CA that there was no calcium in the aggregate).

2. A larger set of analyses for calcium content was made by RAPRA using the method in Appendix III on both the external and internal surfaces of a representative range of fence posts and units. There was a considerable scatter in cement content between individual roof slab samples (extremes were 23 per cent and 18 per cent). Taking into account the non-homogeneity of the samples, the assumption in subsequent calculations of a general cement content of 20 per cent

in the prestressed units was not considered to appreciably affect the validity of the results. A similar treatment of the fence posts gave a mean calcium content of 8.5 per cent in concrete indicating 18.5 per cent cement content.

3. All the chloride in concrete results were then arithmetically converted to equivalent anhydrous calcium chloride in cement using the above average cement contents.

APPENDIX II

ANALYSIS METHOD (from RAPRA Report D 9642)

DETERMINATION OF CHLORIDE ION PENETRATION

PRINCIPLE

In order to study the change of water-soluble chloride ion concentration with depth below the surface of the fire-exposed concrete, test portions were removed from different levels and immersed in water for 24 hours. The concrete was filtered off and the aqueous solution diluted with isopropanol and titrated with mercuric nitrate using a diphenylcarbazone indicator.

PRELIMINARY TESTS

An earlier series of tests using concrete exposed to hydrochloric acid vapour had shown the feasibility of such a procedure.

A preliminary set of successive extractions was carried out to test the efficiency of the extraction of chloride from concrete by water immersion. These showed a significant fraction of the chloride was not extracted in 24 hours from fragments approaching 1 cm in cross-section. This fraction decreased with the size of the fragments and could be ignored for sizes appreciably below 5 mm in cross-section.

EXAMINATION OF CONCRETE BEAMS

FINAL TEST PROCEDURE

Sampling

The beam was cross-sectioned where required using a rotating high-speed carborundum disc, cooling with the minimum amount of distilled water which was applied occasionally as a fine spray.

The black organic deposit on the exposed surfaces was removed by light contact with the side-face of the carborundum disc without removing any significant amount of concrete.

Samples were then taken from the cross-section at successive levels between the two surfaces and the centre of the beam. Cuts up to 1 cm in depth were made in the cross-section between these levels with the carborundum disc, used dry.

The residual ridges were carefully taken out using a cold chisel and the pieces and dust were collected for each sample separately. Each sample was now crushed to particles smaller than 2 mm diameter using a pestle and mortar with a cover to prevent loss.

Determination of chloride content

A representative test portion taken from the crushed sample was weighed, 25 ml of distilled water was added and left for 24 hours. The solution was then filtered into a 100 ml graduated flask and the flask containing the concrete washed in turn with 5-10 ml water and 50 ml isopropyl alcohol. The combined solution and washings were made up to 100 ml with isopropyl alcohol and the flask shaken well. 50 ml was then pipetted into a titration flask, 10 ml isopropyl alcohol were added, the pH adjusted with nitric acid until indicated acidic by bromocresol green and the solution titrated with 0.01N. mercuric nitrate solution (standardised against sodium chloride) after the addition of diphenylcarbonazone indicator. A blank correction was made before calculation of the chloride content.

APPENDIX III

DETERMINATION OF CALCIUM AND MAGNESIUM CONTENTS OF CONCRETE SAMPLES BY RAPRA (from RAPRA Reports 10463 and 10573)

1. PREPARATION OF SAMPLES

Any black organic surface deposit was removed and the requisite amount of material was cut from less than 6 mm ($\frac{1}{4}$ in) below the exterior face of each concrete section using an abrasive disc and broken up with a pestle in an enclosed mortar. The crushed material was passed through a 50 mesh sieve and, after determining the proportions of coarse and fine material, two samples were made up by recombining the coarse and fine particles in the correct proportions to provide reliable duplicates. The above procedure was carried out on the fence post and prestressed unit and one sample from each was sent to the Cement and Concrete Association for a detailed analysis.

2. CHLORINE DETERMINATION

Duplicate, weighed 3 g samples of the prepared concrete (homogenised by tumbling) were extracted with 25 ml water for 24 hours at room temperature. The liquid was decanted through a small filter paper and the extraction repeated for a further 24 hours with 15 ml water. This was decanted through the same filter paper (which had been stored in a room free from acid/alkali contamination), followed by two washings of the residue. The combined filtrates were made up to standard volume and two equal portions from each were titrated with standard mercuric nitrate solution.

3. CALCIUM AND MAGNESIUM DETERMINATION BY ATOMIC ABSORPTION

From each of the prepared concrete samples (para.1) accurately weighed samples (0.5 g) were boiled with a little distilled water containing 2 ml 5 N hydrochloric acid. The solutions were filtered and made up to 500 ml with water. These solutions represented a sample concentration of 1000 ppm. This having been done, absorptions at the calcium and magnesium wavelengths were recorded using a Hilger Atomspek absorption spectrophotometer with both air/acetylene flame and nitrous oxide/ acetylene flame.

PLATE 1. SUPERIMPOSED LOAD ON THE CONCRETE UNITS IN THE FORM OF FLETTON BRICKS

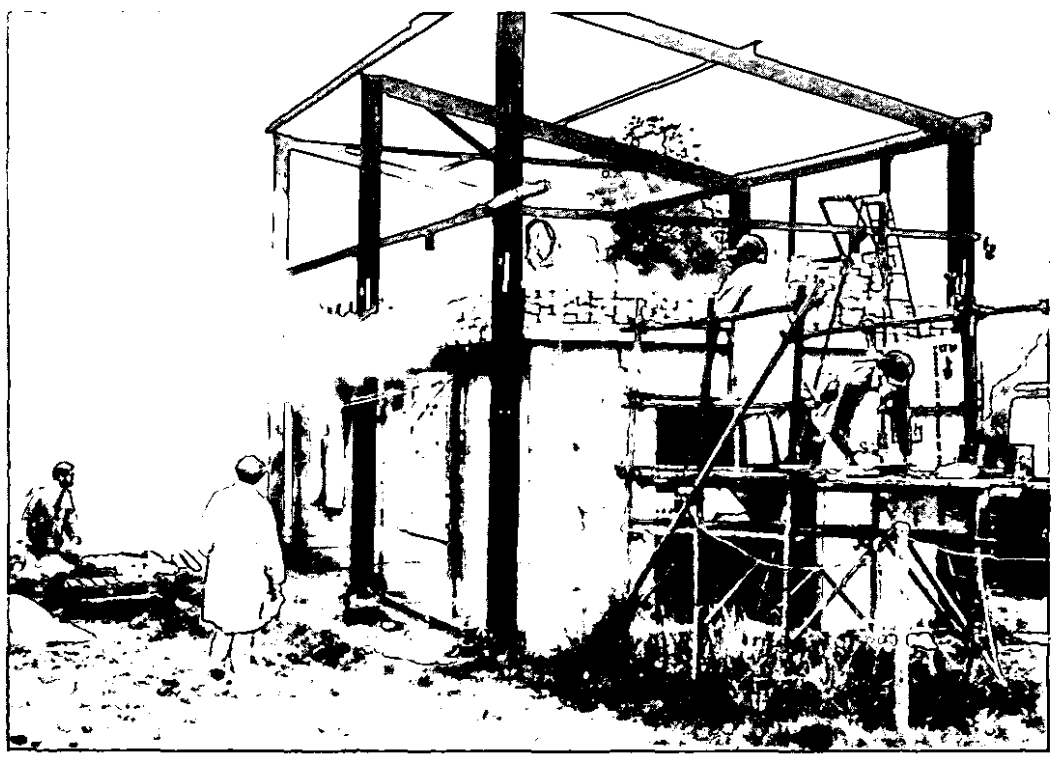


PLATE 2. GENERAL VIEW OF THE BUILDING DURING TEST

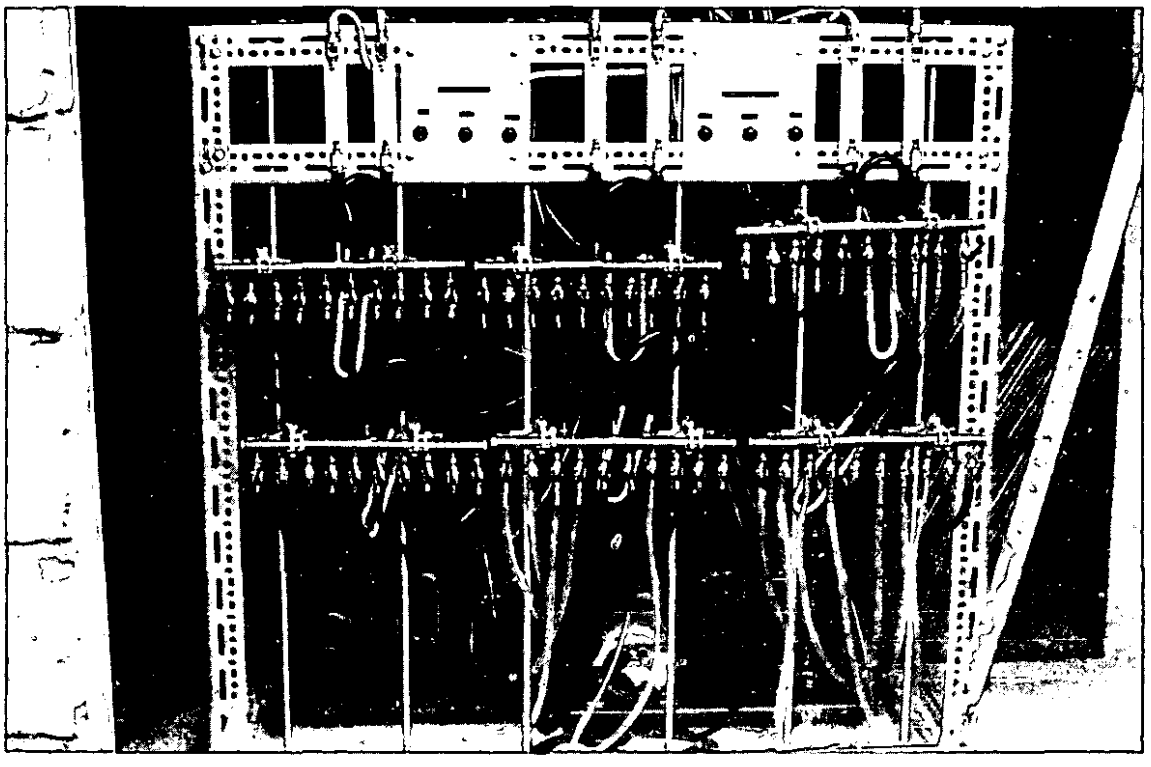
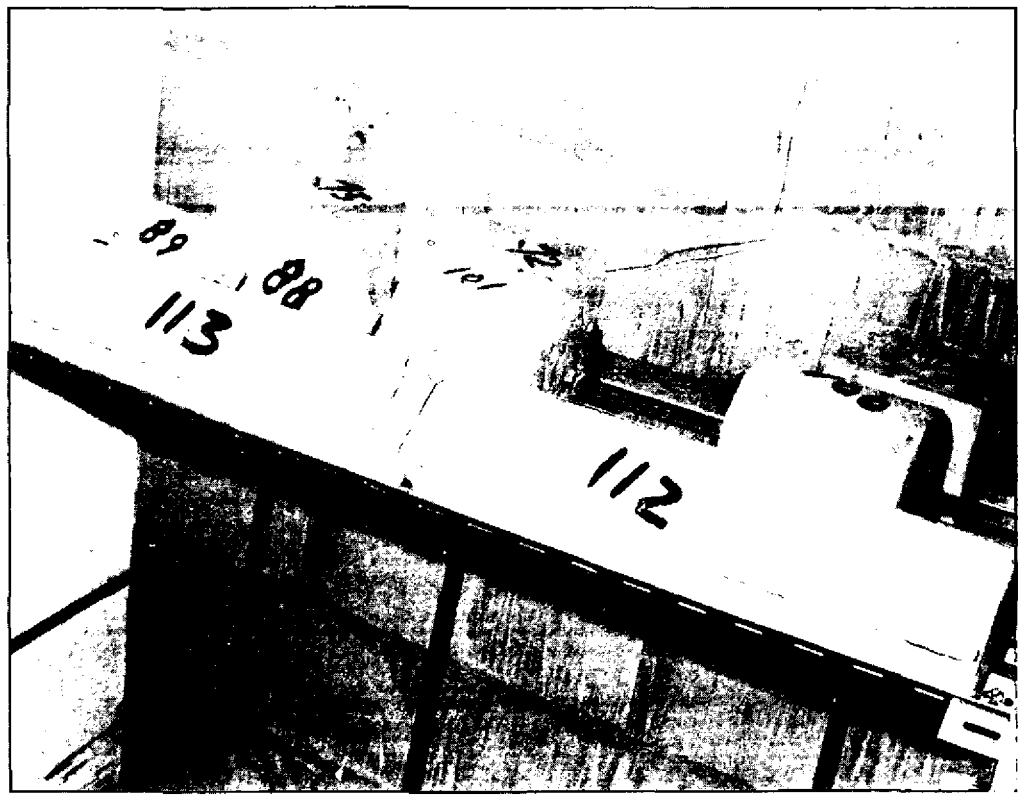
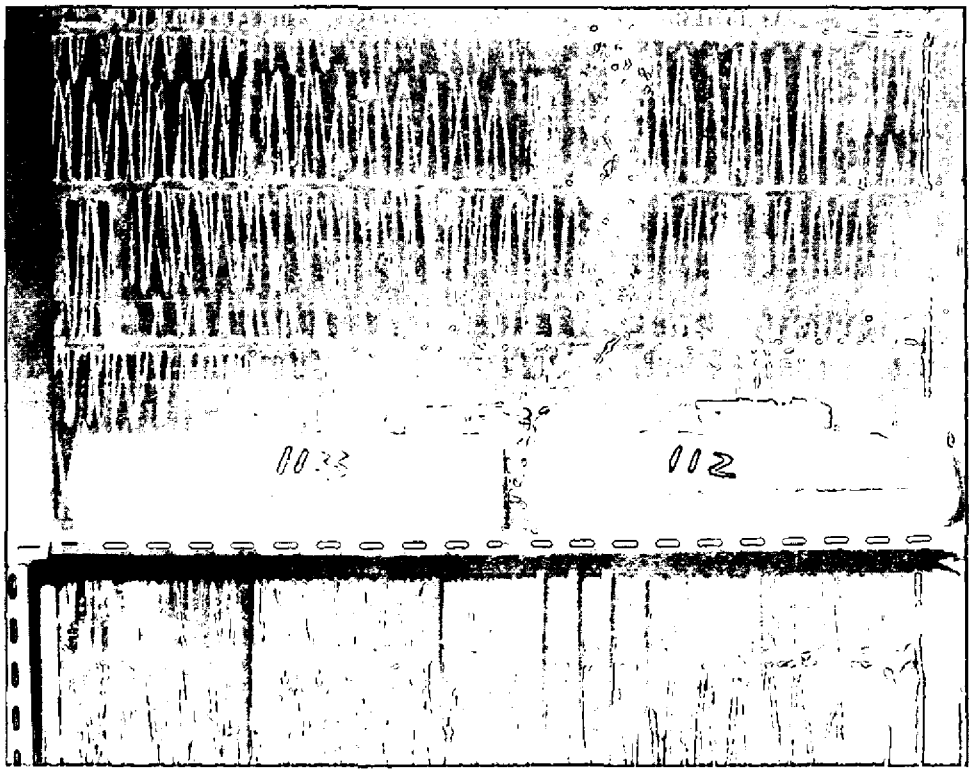


PLATE 3. GAS SAMPLING EQUIPMENT



PLATES 4 and 5. CONCRETE SAMPLES MOUNTED ON STANDS - BEFORE AND AFTER TEST



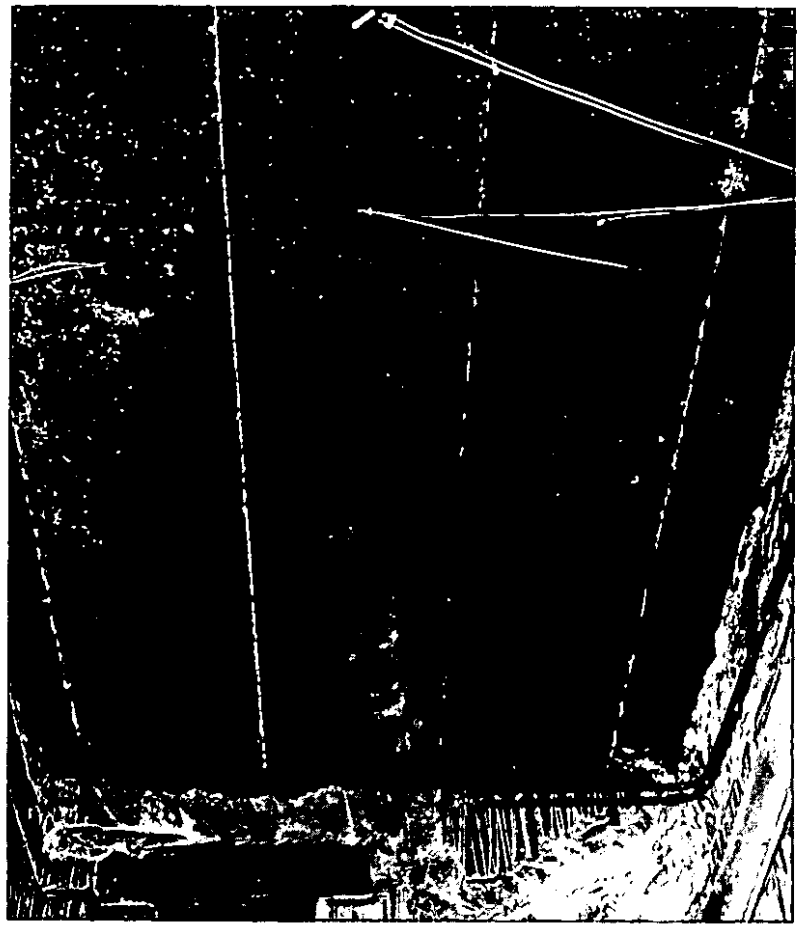


PLATE 6. THE APPEARANCE OF ROOF UNITS AFTER TEST

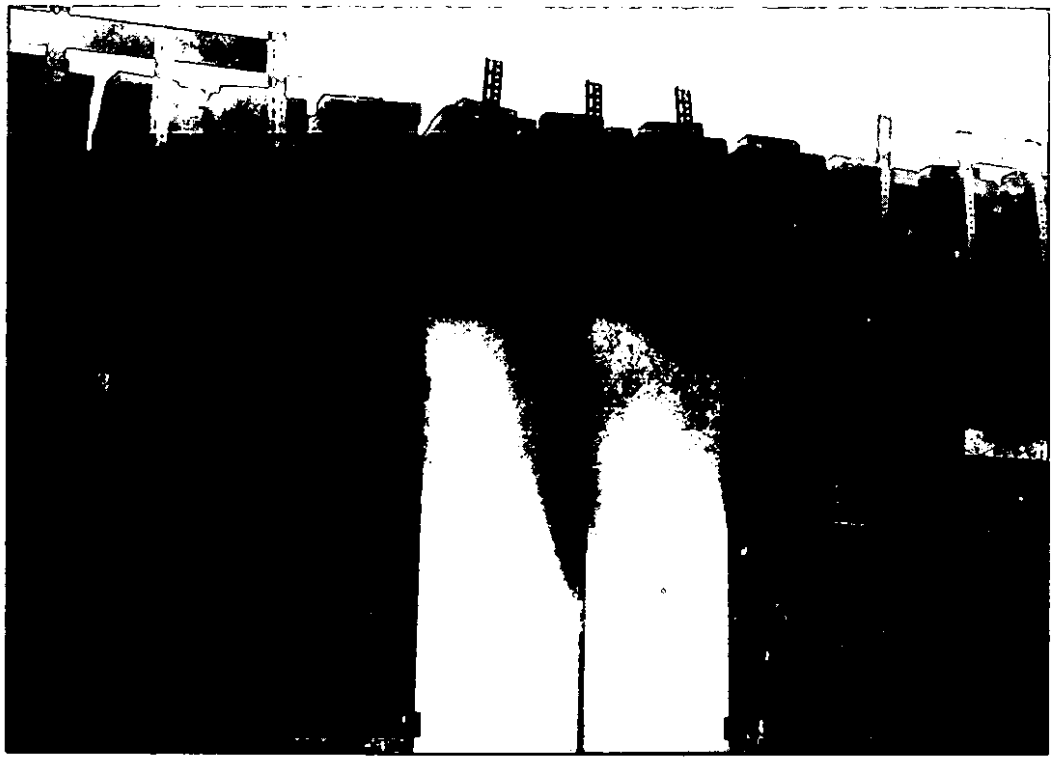


PLATE 7. THE THREE FIRE CHAMBER ROOF UNITS DEFLECTING DURING A TEST

PLATE 8. ROOF UNIT SUBJECTED TO STRUCTURAL TESTING

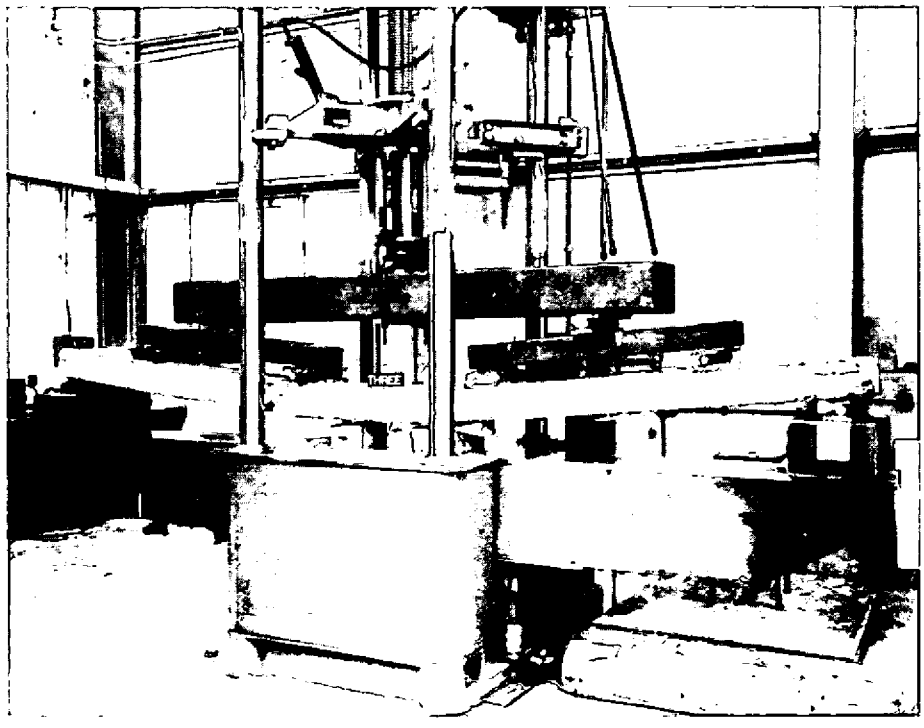


PLATE 9. CONTROL ROOF UNIT UNDERGOING HYDRAULIC LOADING

PLATE 10. PHENOLPHTHALEIN TEST APPLIED TO A FRESHLY BROKEN CONCRETE CUBE WITH A STEEL ROD PASSING THROUGH IT.

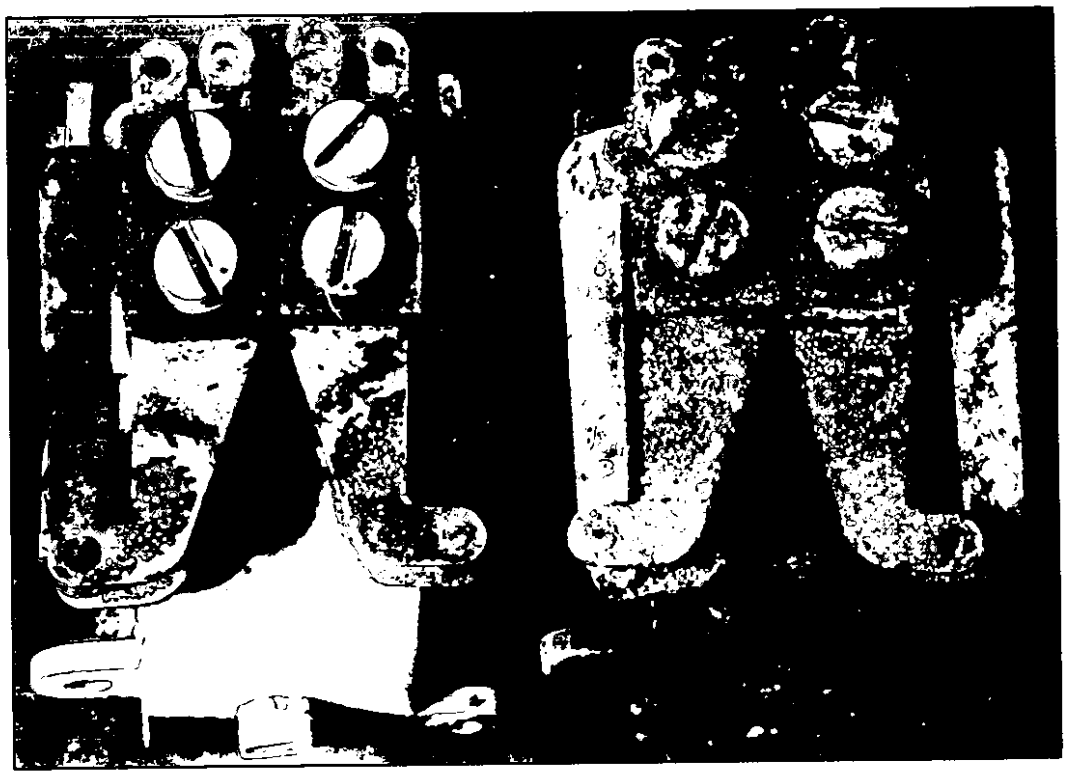


PLATE 11. WASHED AND UNWASHED RELAYS AFTER 6 MONTHS HUMID STORAGE

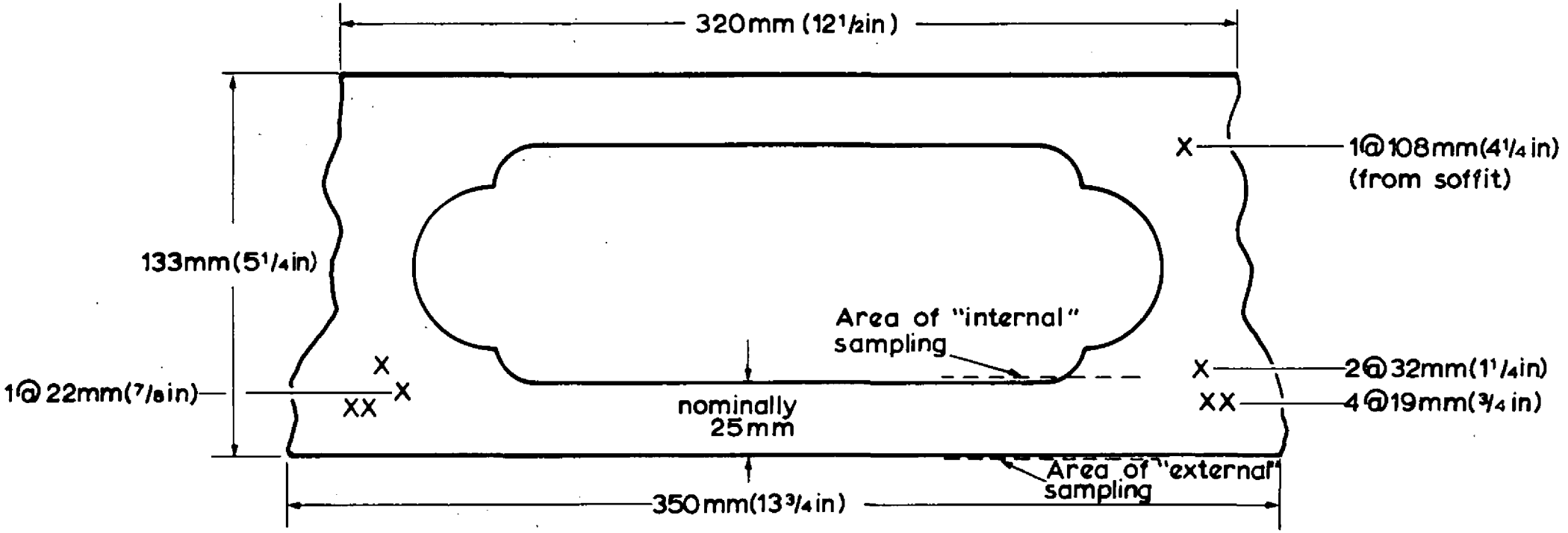


Figure 1 Section through 133mm (5¼in) prestressed concrete floor units showing manufacturers stated positions of 8 No 10 SWG wires

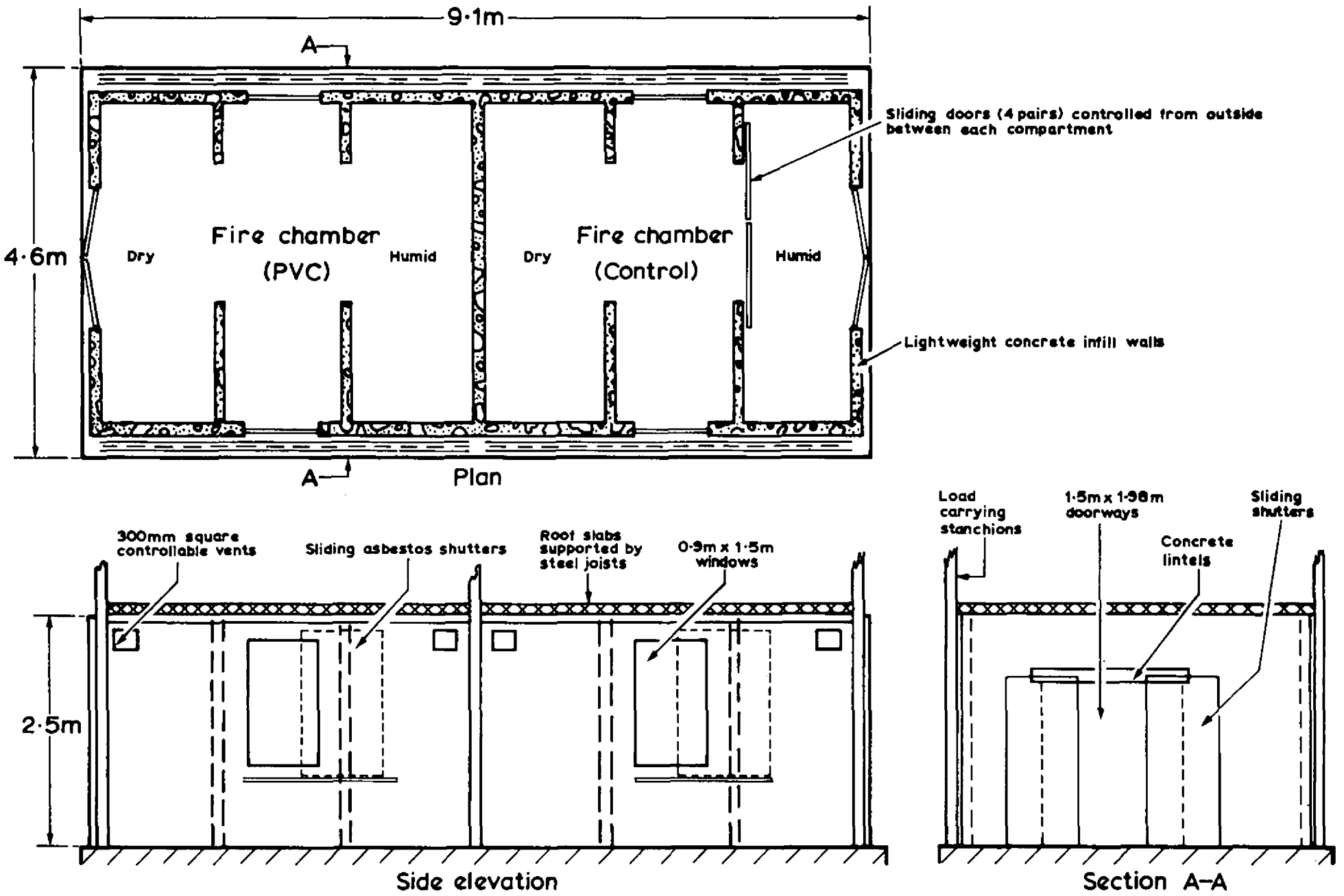


Figure 2 Test building

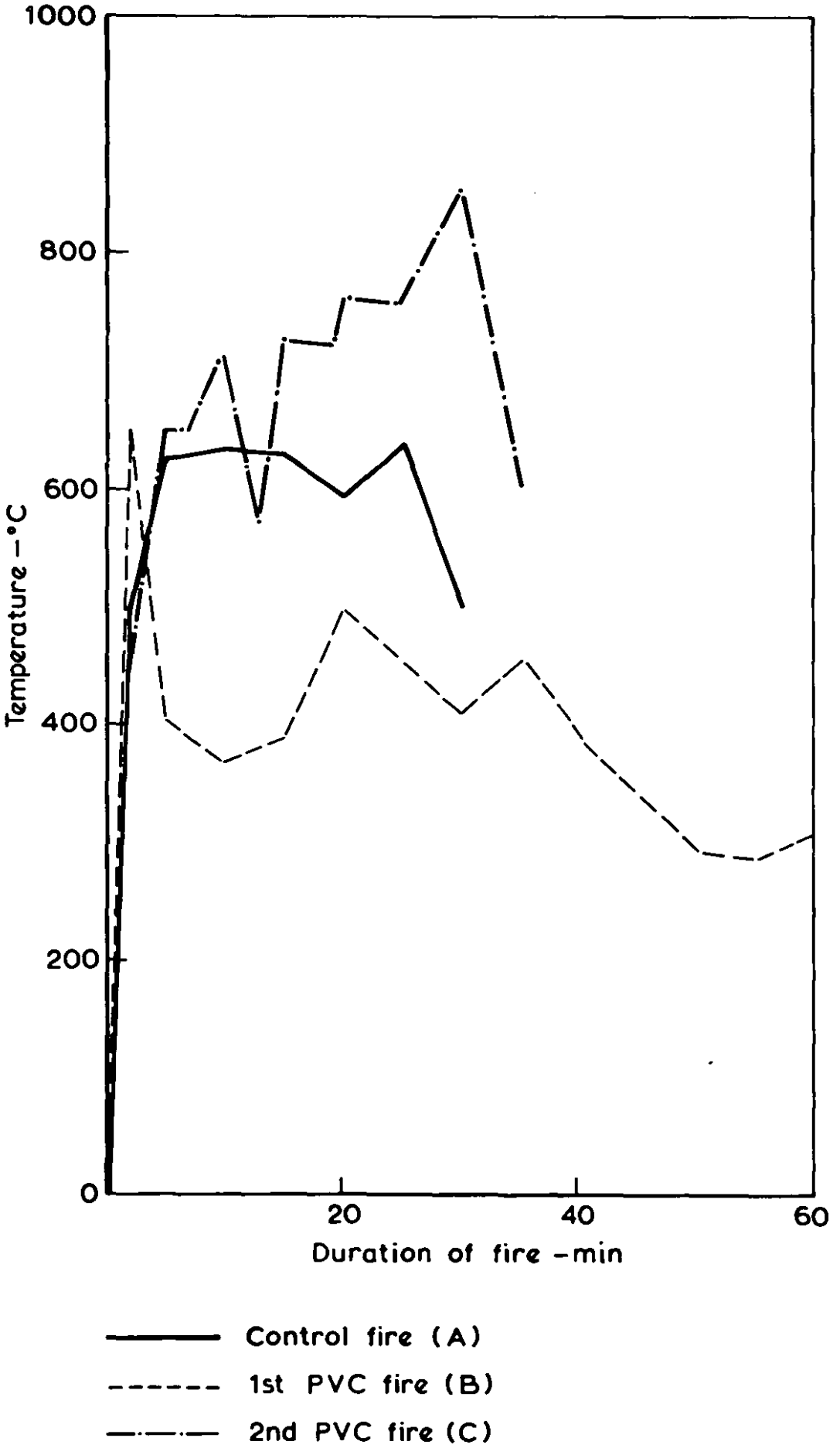


Figure 3 Mean air temperatures in the fire compartments

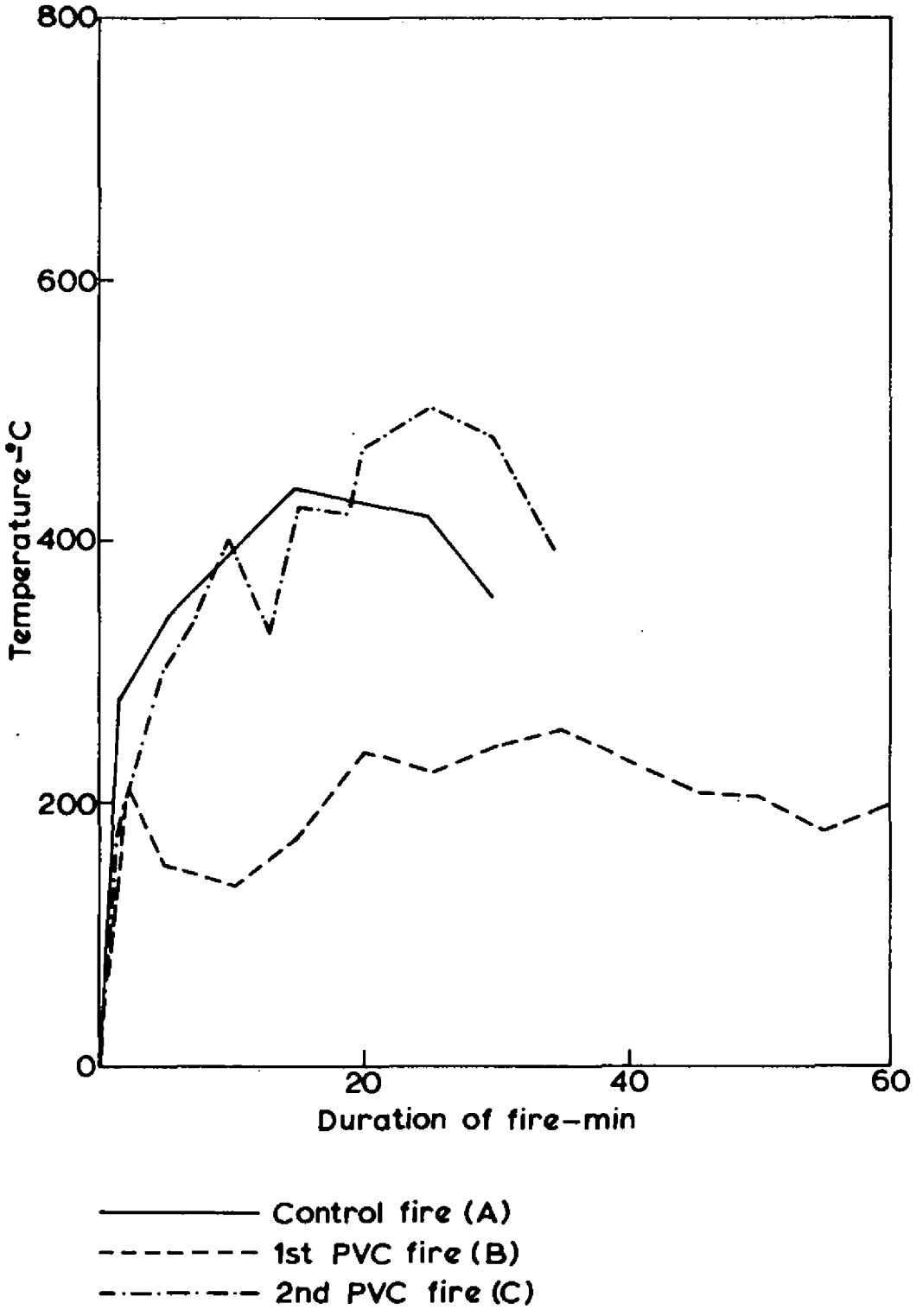


Figure 4 Mean air temperatures in the humid compartments

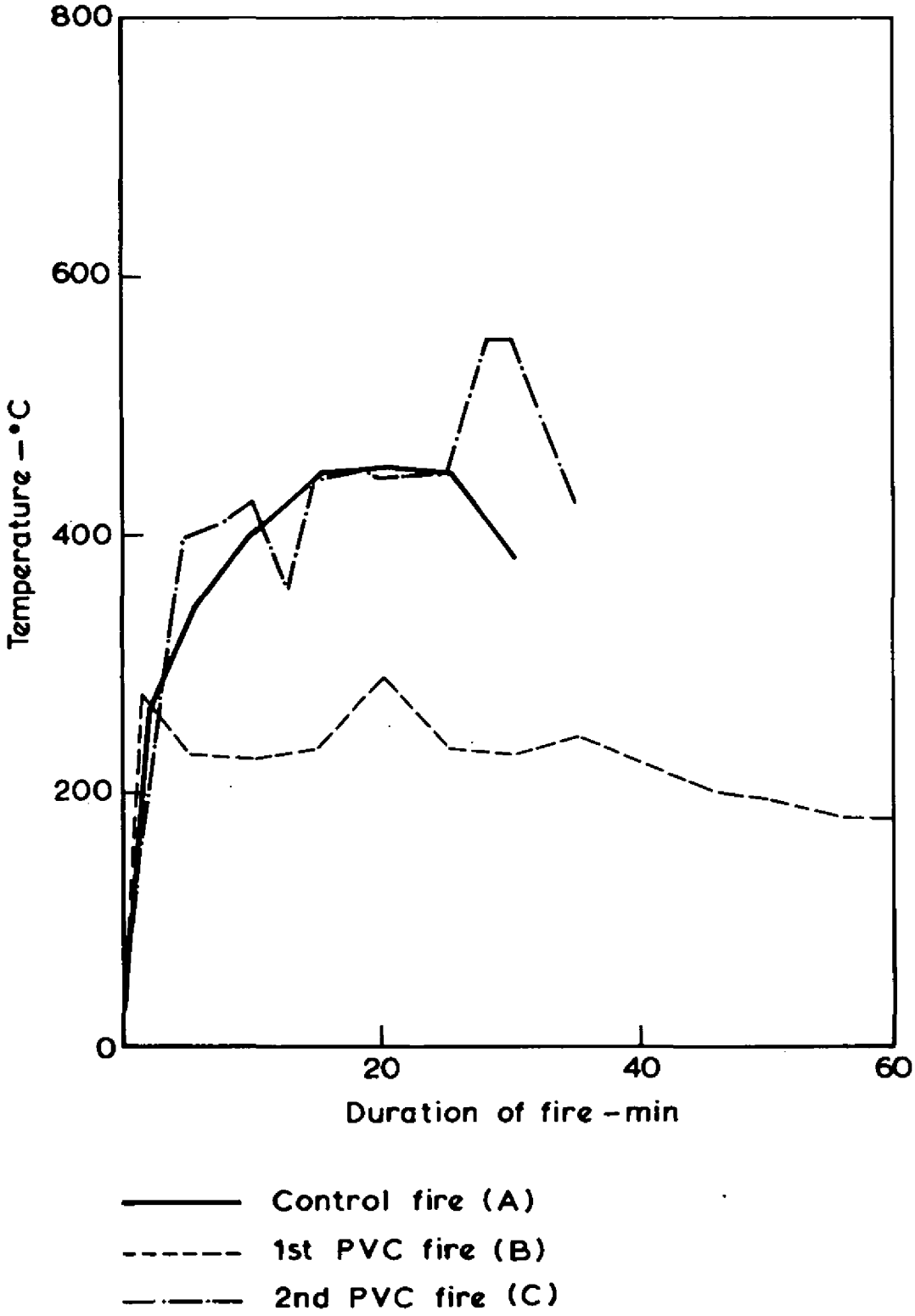


Figure 5 Mean air temperatures in the dry compartments

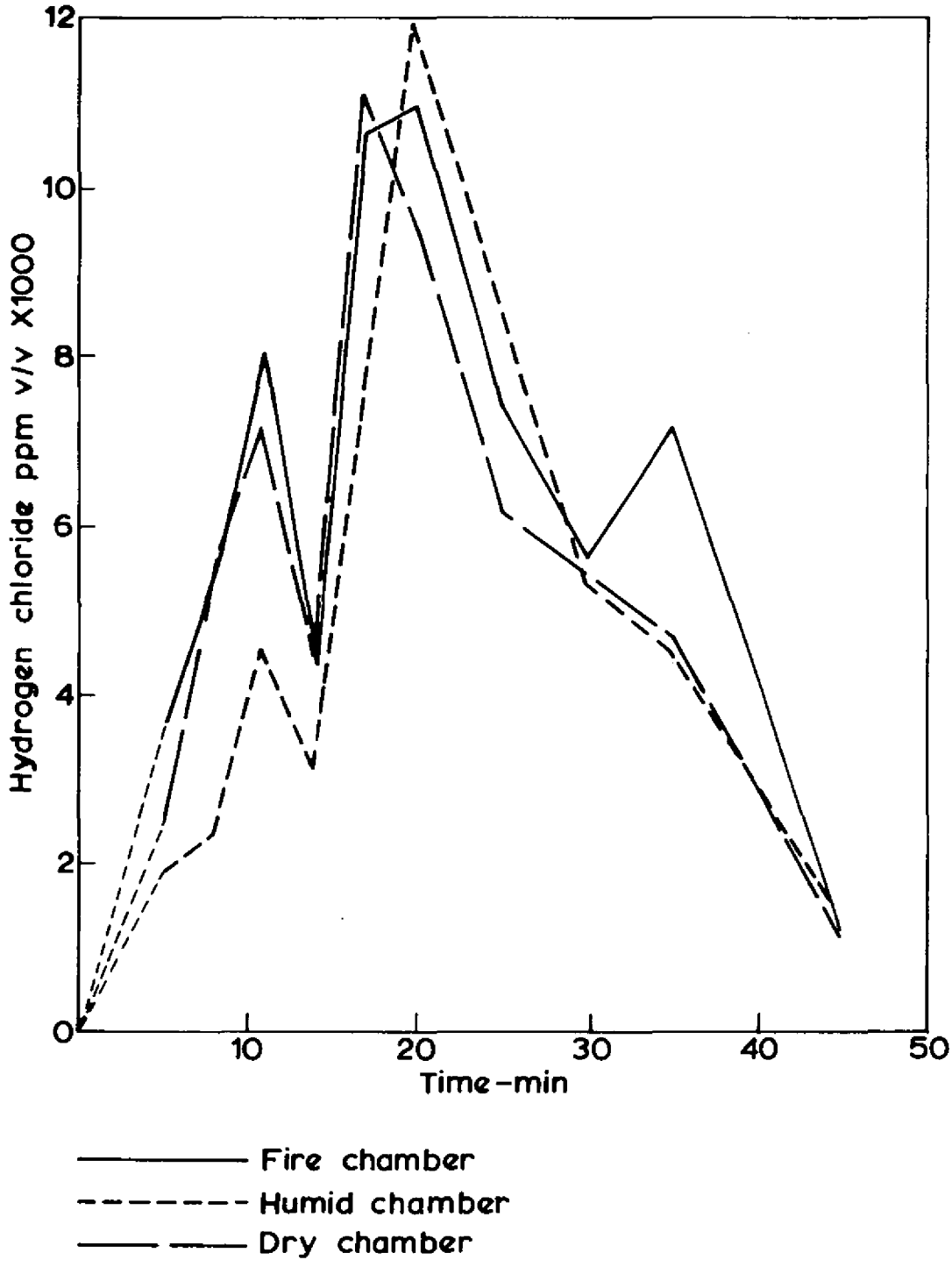


Figure 6 Hydrogen chloride concentrations
-test C

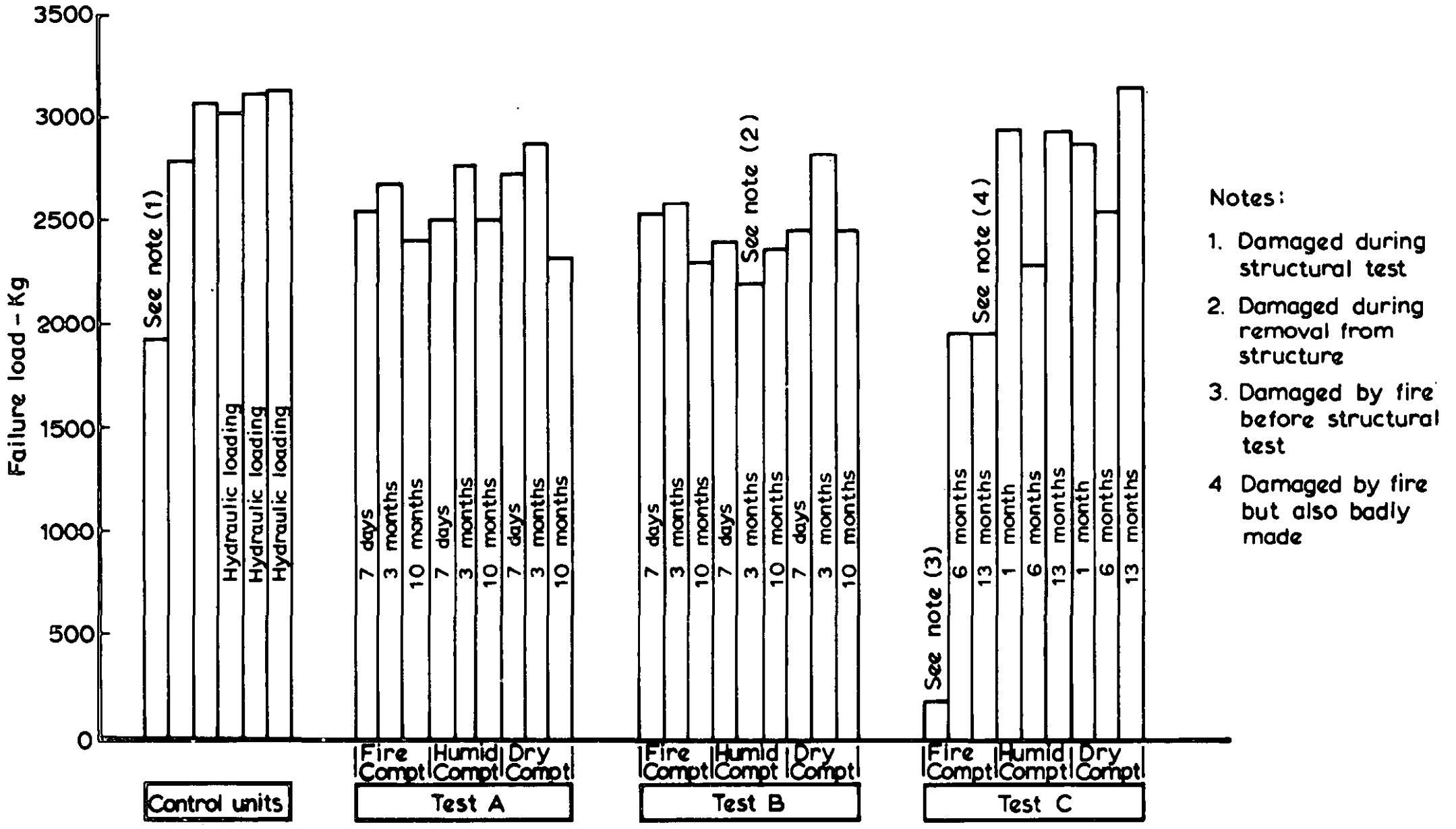


Figure 7 Results of loading tests on prestressed units

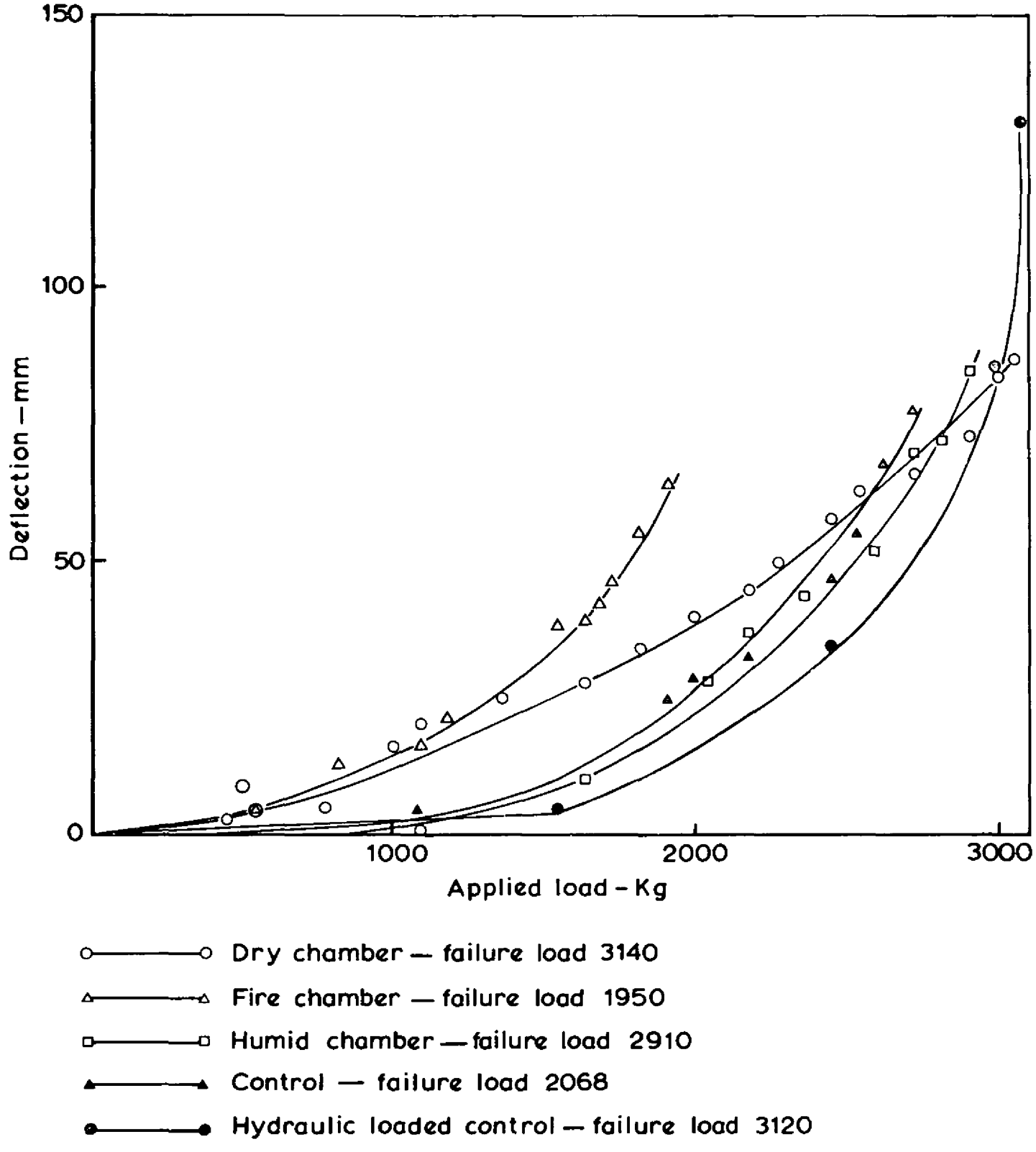


Figure 8 Test C Deflection of prestressed units removed after 13 months

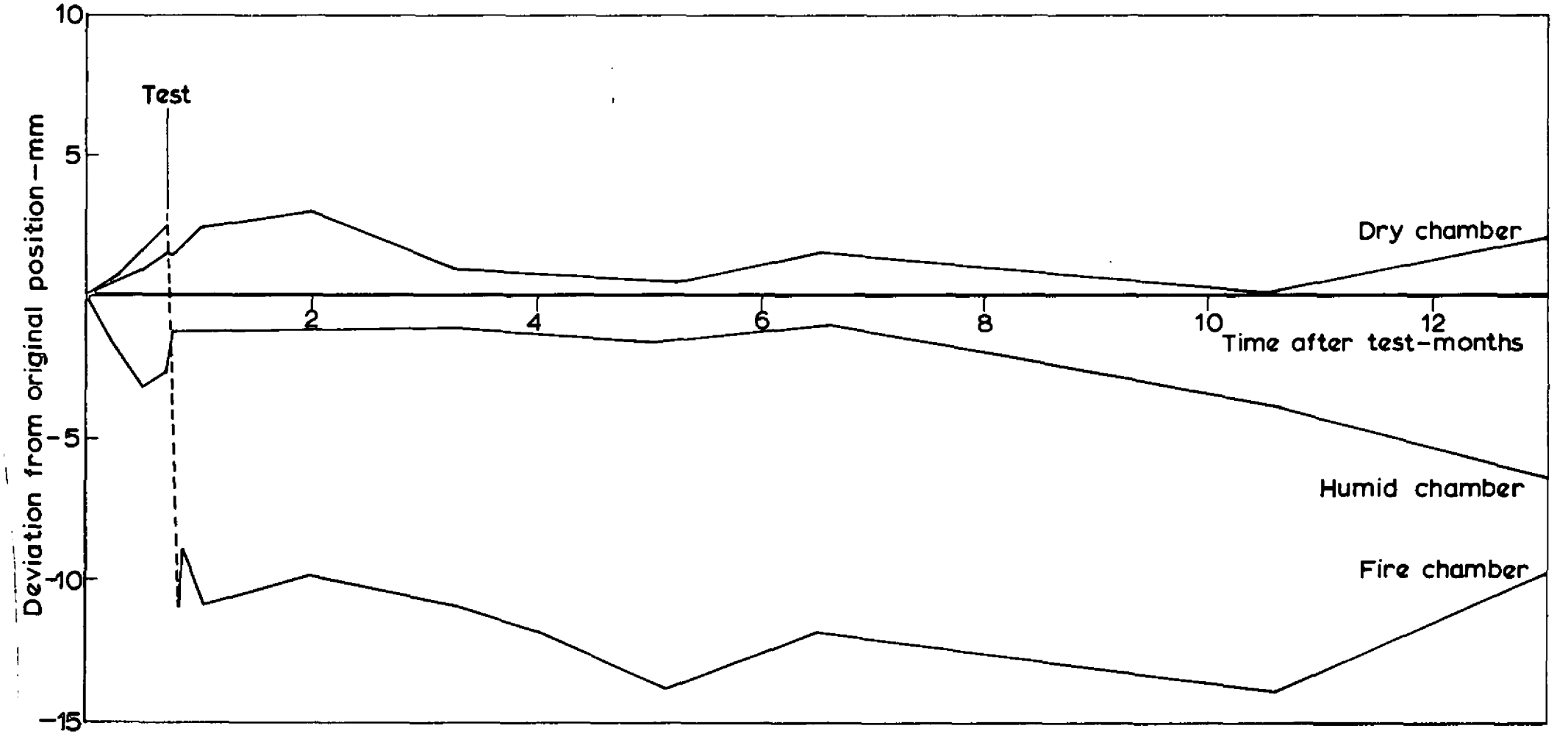


Figure 9 Deflection of prestressed units remaining on building for 13 months – test C

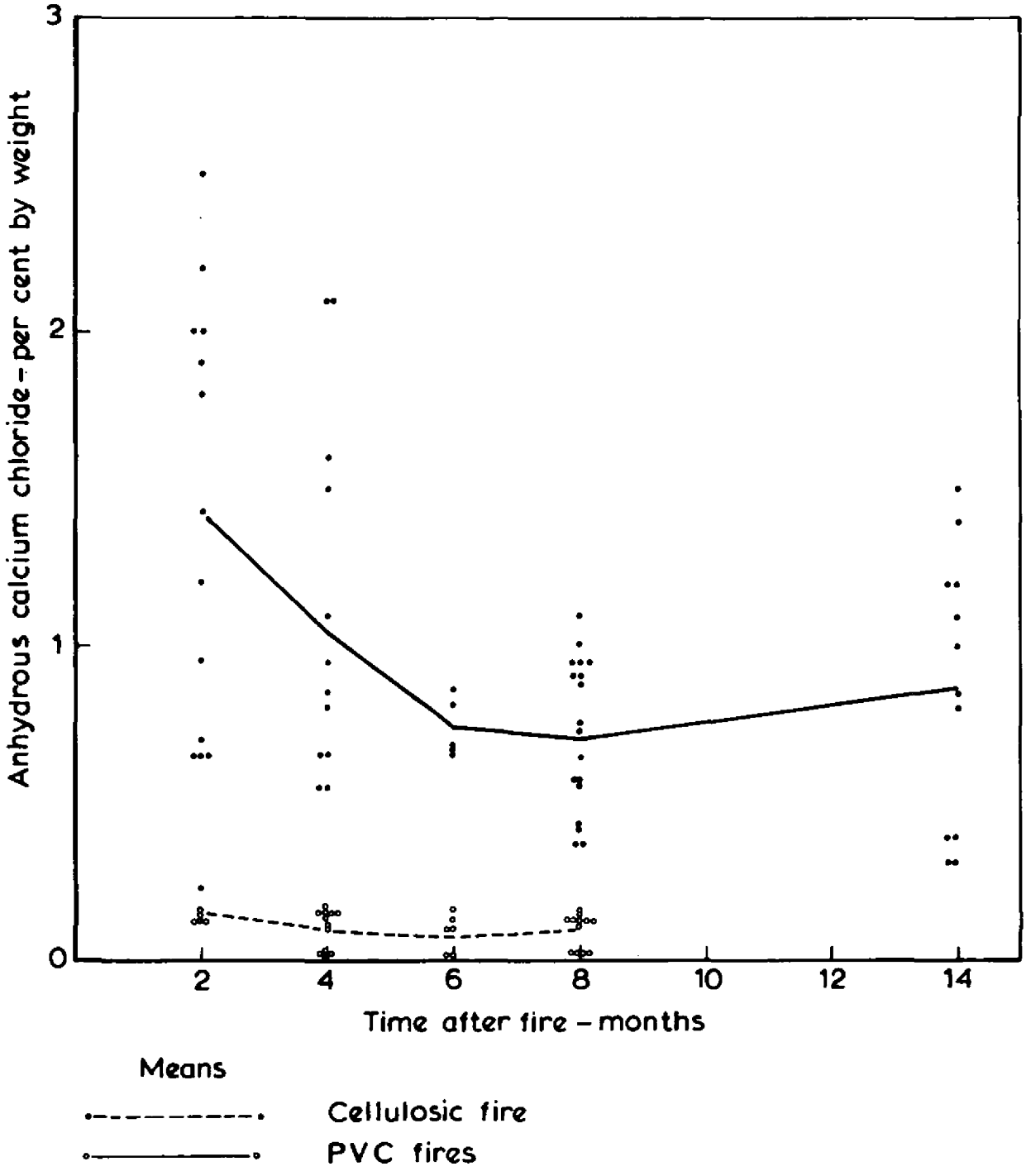
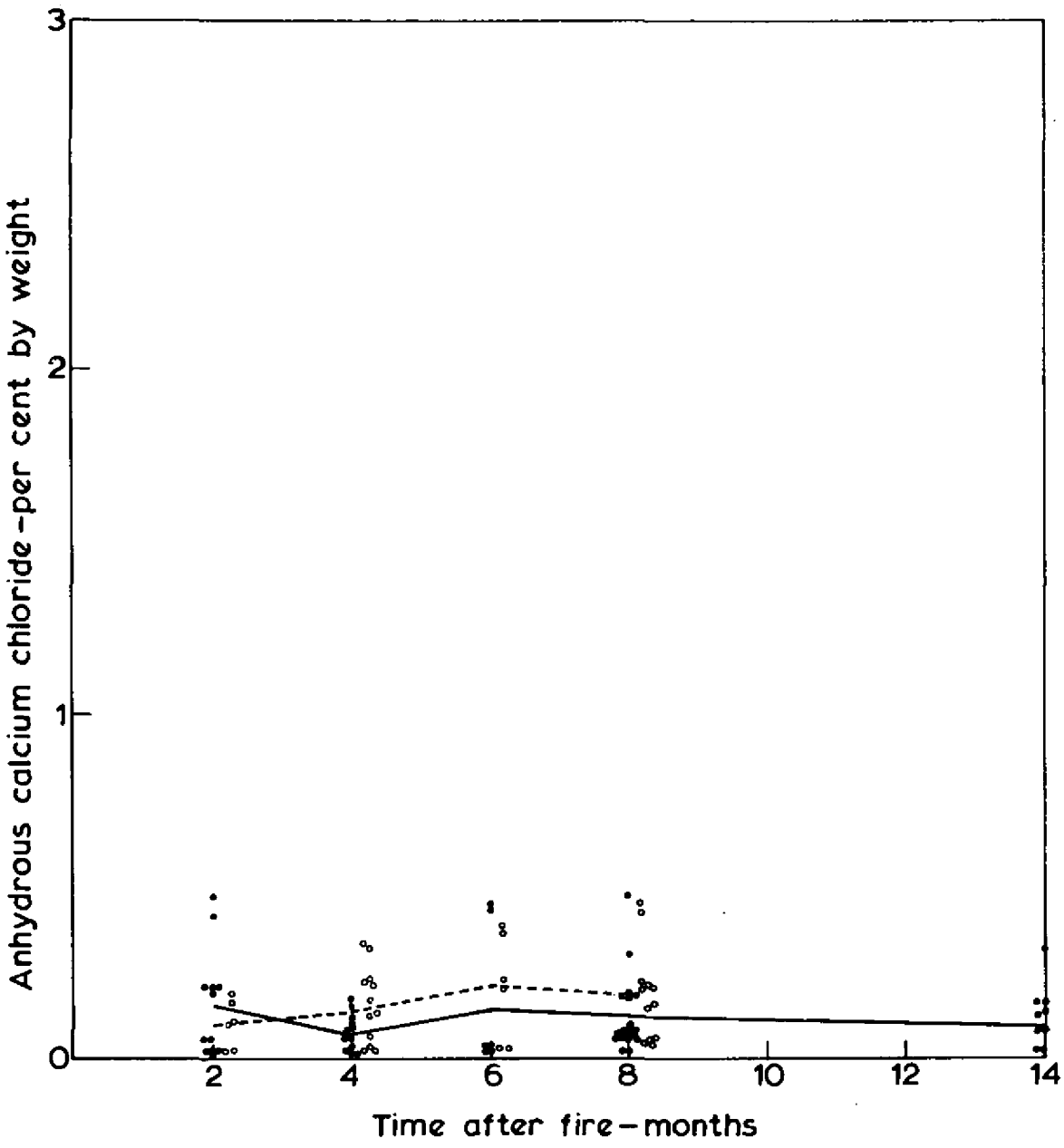


Figure 10 Chloride concentrations – Exposed surfaces of prestressed units



Mean values
∘--------∘ Cellulosic fires
•———• PVC fires

Figure 11 Chloride concentrations-
internal surfaces prestressed units

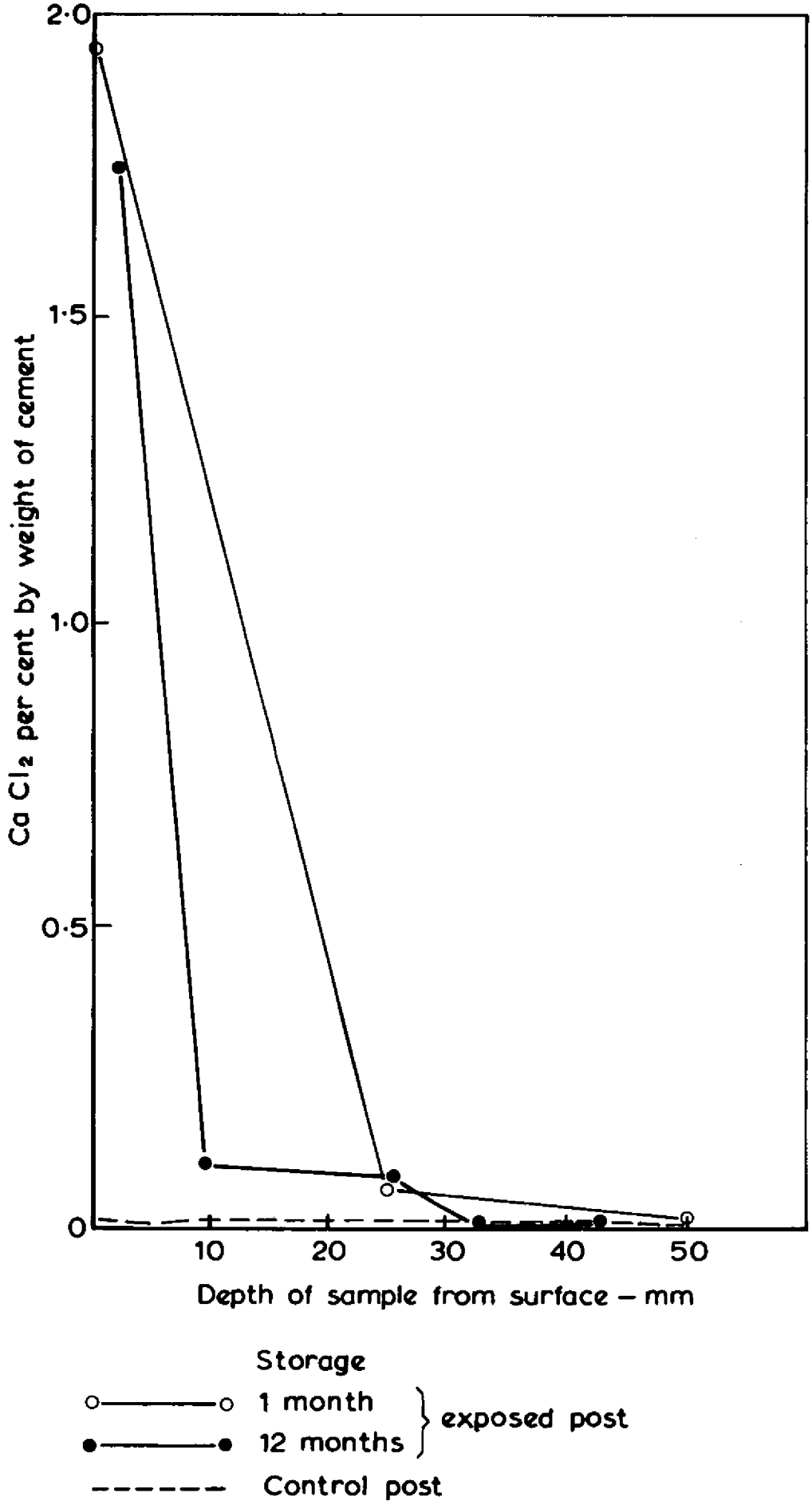


Figure 12 Chloride concentrations - fence posts

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