

Improving Precision of Rubber Test Methods: Part 2 — Ageing

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ABSTRACT

Many Interlaboratory Test Programs, ITPs, on rubber test methods, have been carried out during the last years. ITPs have been organized within ISO TC 45 and also in several countries such as USA, UK and Sweden. Most of these ITPs have shown that the repeatability and the reproducibility are poor for many rubber test methods.

In an attempt to do more than just determine the poor precision, we chose four methods and decided to study them to identify the factors giving poor precision, thereby improving the methods.

This second part contains the work done on heat ageing tests. The results from the project show the temperature and air speed to be the most critical factors. A good ageing oven must therefore have accurate temperature in time and space, low air speed dependent on the air exchange rate only and means of controlling the air exchange rate.

1 BACKGROUND

At the beginning of the 1980s it was decided to include within ISO TC 45, Rubber and Rubber Products, a precision clause in all testing method standards. The precision clauses were established by carrying out Interlaboratory Test Programs, ITPs, to establish the *repeatability* (within laboratories) and the *reproducibility* (between laboratories) for the test methods.

In 1981 the ISO published a standard for determination of the precision of test methods, ISO 5725-86.¹ In 1984 TC 45 published a technical report, ISO TR 9272,² for guidance on how to establish precision data for rubber test methods. ISO TC 45 has since then carried out about 25 interlaboratory test programmes.

This work inspired us in Sweden to start an interlaboratory test programme, organized by the Swedish National Testing Institute. During the years 1982-88, 14 interlaboratory tests were carried out. For two of the methods a retest was done. Up to 25 laboratories participated in these interlaboratory tests.

All these interlaboratory tests within the ISO and in Sweden have shown that the spread in the test results is worse than anyone could have expected. At the same time, the requirements for the products have increased, which means that we need to be able to test the properties of rubber materials with a higher accuracy than before. It must not be the case that what we measure mainly reflects the spread in the testing and does not show the variations in the material tested.

2 THE PURPOSE OF THE PROJECT

The purpose of the project was to achieve a lower spread in test results, within and between laboratories, for the test methods under study. The results from this project will be presented to the Swedish Standards Institution and to the ISO as a basis for improving the test method standards. This project was started in 1989.

3 PARTICIPATING COMPANIES

The following companies have participated and financed this project:

- Alfa-Laval Materials AB
- Forsheda AB
- Horda Compound AB
- Skega AB
- Statens Provningsanstalt
- Sunnex AB
- Trelleborg Industri AB
- Viskafors AB
- Volvo Flygmotor AB
- Volvo PV AB, materiallab
- Värnamo Gummifabrik AB**
- Saab-Scania AB, Scaniadivisionen

4 THE ORGANIZATION OF THE PROJECT

4.1 General organization

The following methods have been studied during the project:

- hardness, normal and micro IRHD, according to ISO 48³ and Shore according to ISO 7619⁴
- tensile test, according to ISO 37⁵
- heat ageing, according to ISO 188⁶
- temperature retraction test, TR, according to ISO 2921⁷

Good background was obtainable for all of these methods as all of the tests have been studied one or more times by interlaboratory trials.

The test methods have been studied by investigating the influence of different factors on the spread in test results.

At the beginning of the project a visit was paid to all participating companies to make up an inventory of the type of test instruments that are being used. Some preliminary interlaboratory tests and other measurements were also made.

Before starting the project a literature search was performed (1990), but we found very little published about precision of rubber testing.⁸⁻¹⁰ Further papers have been published after 1990.¹¹⁻¹⁴

4.2 Organization of the heat ageing part

The project started with an inventory of the ovens used for the ageing tests among the 12 participating Swedish rubber companies. Based on this inventory four of the ovens and a new designed oven were chosen for a closer study. The ovens were investigated for the following:

- temperature uniformity in time
- temperature uniformity in space
- set, shown and actual temperature
- air speeds
- air exchange rates
- ageing results in different ovens

The following factors influencing the ageing were then investigated in different ways:

- temperature
- air speed

5 HEAT AGEING RESULTS

5.1 Inventory of ovens for heat ageing

The equipment used for ageing by the participating laboratories is shown in Table 1. Elastocon, Wallace and Scott ovens are specially designed for ageing of polymer materials, the other ovens are ordinary laboratory shelf ovens.

5.2 Measurements of ageing ovens

Based on the inventory the following ovens were measured in one laboratory:

- Heraeus UT 5042
- Heraeus UT 5060E
- Salvis TSW 60
- Elastocon EB 01 (cell oven)
- Elastocon EB 04 (new type of ageing oven)

Elastocon EB 01 is a cell oven and the others are shelf ovens. EB 04 is a new type of shelf oven specially designed for the ageing of polymer materials.

TABLE 1
Equipment Used for Heat Ageing

Company	Shelf ovens				Cell ovens		
	Heraeus	Salvis	Memmert	Other	Elastocon	Wallace	Scott
1	5		6	—	1	—	1
2	7		—	—		—	—
3	2	—	—	6	3	—	—
4	1	12		—	2	—	—
5	7	—		—	—		—
6	17	—	—	—	—	—	
7	12	45	5	—	4	9	—
8	—	—	3	—	1	—	—
9	8	—	—	5	1	—	2
10	—	6	—	—		—	—
11	27		—	—	—	—	—
12	3	—	—	—	1	—	—
13	2	1	1				
14		—		—	1	—	—
Sum	91	64	15	11	14	9	3

5.2.1 Temperature variations in time

A PT 100 sensor was placed in the centre of each oven. The sensors were connected to a data logging system connected to a PC computer, with a resolution of 0.1°C. The temperature was adjusted as close as possible to 100°C. The ovens were run for 5 days to see temperature variations in time. The results are shown in Table 2.

The UT 5042 oven had a mechanical controller, the other ovens had electronic controllers. With a modern electronic controller temperature variations in time seem to be no problem.

5.2.2 Temperature variations in space

A frame with five PT 100 sensors located in each corner and in the centre, connected to a data logging system, was placed inside each cabinet and was moved between three positions. The outer sensors were placed about 50 mm from the walls. The results are shown in Table 3 and Figs 1-5. The table shows the difference between five points in each location and the total difference (all points all locations).

5.2.3 Set, shown and actual temperatures

It is not only the temperature variations in time and space that are important, but also to know the actual temperature in the oven. To check

TABLE 2
Temperature Variations in Time

<i>UT 5042</i> (°C)	<i>UT 5060E</i> (°C)	<i>TSW 60</i> (°C)	<i>EB 01</i> (°C)	<i>EB 04</i> (°C)
13.8	0.1	0.2	0.1	0.1

TABLE 3
Temperature Variations in Space

<i>Location</i>	<i>UT 5042</i> (°C)	<i>UT 5060E</i> (°C)	<i>TSW 60</i> (°C)	<i>EB 01</i> (°C)	<i>EB 04</i> (°C)
Inner	0.9	0.5	1.3	NA	0.4
Center	0.7	1.7	1.3	NA	0.3
Outer	0.7	1.1	2.7	NA	0.2
Total	1.2	1.7	3.1	0.8	0.4

NA = not applicable.

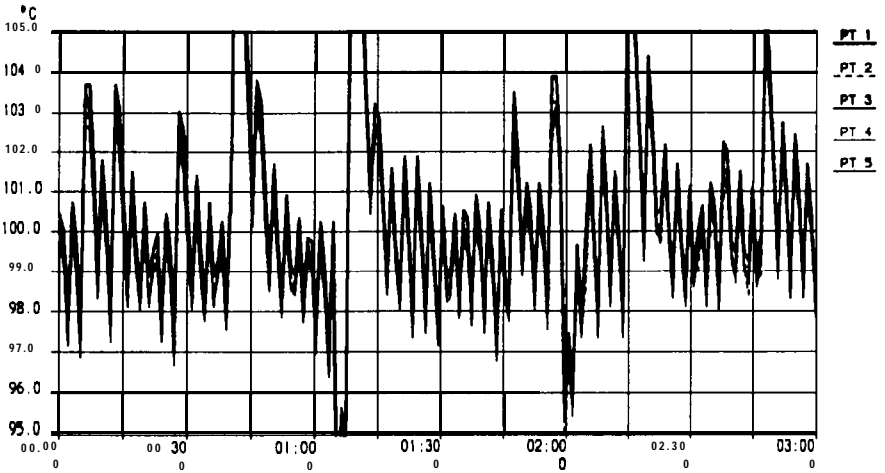


Fig. 1. Temperature variations in space, UT 5042.

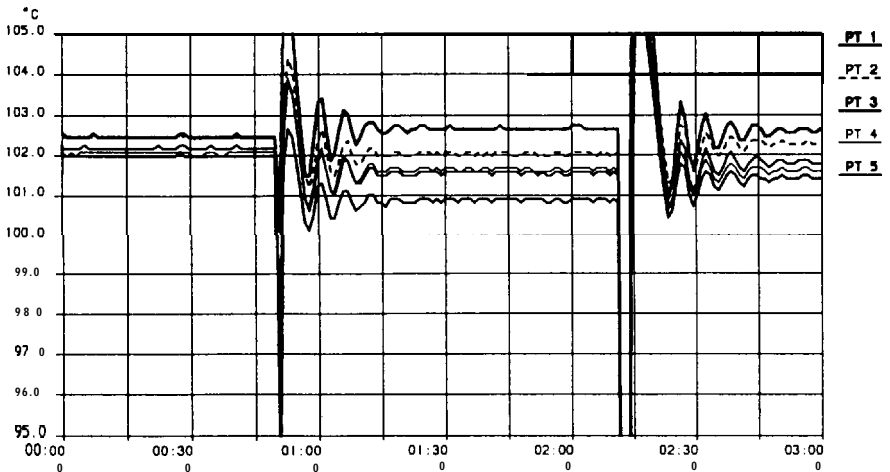


Fig. 2. Temperature variations in space, UT 5060E.

this PT 100 sensors were placed in the centre of each oven. The temperatures were adjusted to 100°C in the centre of each oven and the set and shown temperatures for the ovens were noted, see Table 4.

In the ISO 188 rubber-accelerated ageing test, the temperature tolerances are $\pm 1^\circ\text{C}$ up to and including 100°C, $\pm 2^\circ\text{C}$ up to and including 200°C and $\pm 3^\circ\text{C}$ above 200°C.

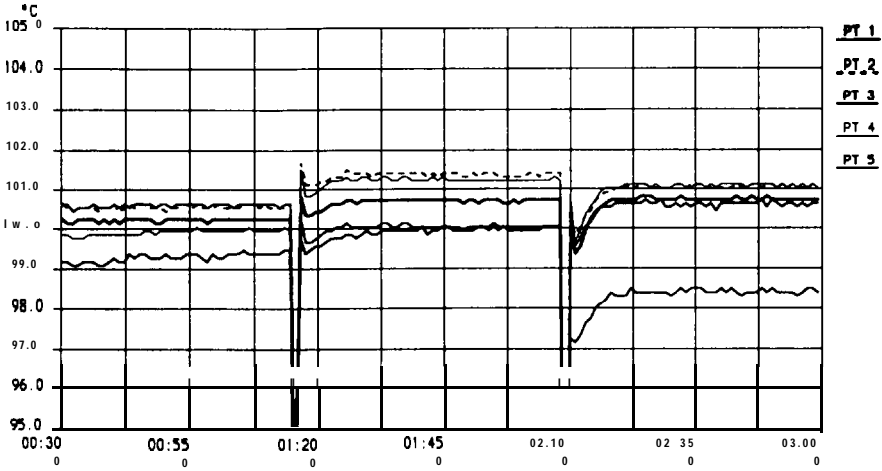


Fig. 3. Temperature variations in space, TSW 60.

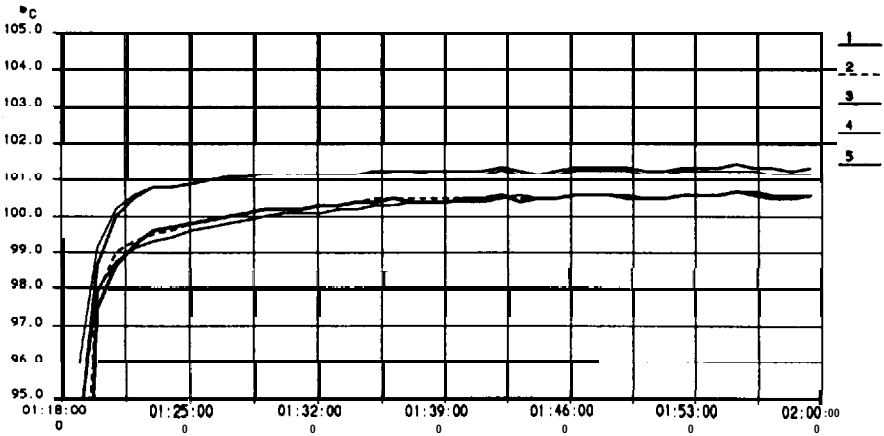


Fig. 4. Temperature variations in space, EB 01.

5.2.4 Air speeds

The air speeds have been measured at 27 points in the ovens and the results are shown in Table 5. Figure 6-8 show the air movement and speeds in the centre plane of each oven. The air speeds are in m/s. The speeds are measured 50 mm from the walls of the cabinet.

The air speeds of ovens EB 01 and EB04 are dependant on the air exchange rate only and it has not been possible to measure them. The value is calculated from the air exchange rate.

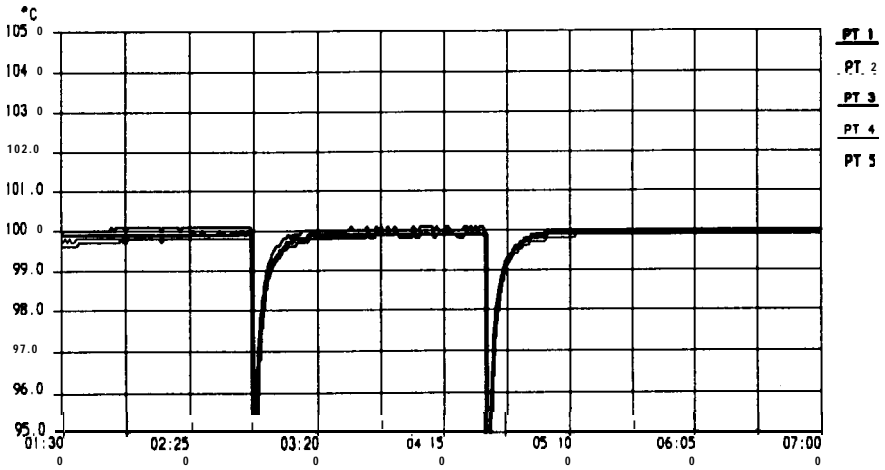


Fig. 5. Temperature variations in space, EB 04.

TABLE 4

Set, Shown and Actual Temperatures

Temp.	UT 5042 (°C)	UT 5060E (°C)	TSW 60 (°C)	EB 01 (°C)	EB 04 (°C)
Set	99	97.5	97.2	100.1	100.0
Shown	96-104	97.5	96	100.1	99.9
Actual	96-103	100.0	100.0	100.0	100.0

TABLE 5

Air Speeds

Speed	UT5042 (m/s)	UT 5060E (m/s)	TSW60 (m/s)	EB 01 (m/s)	EB 04 (m/s)
Min. speed	0.5	0.0	0.4	<0.001	<0.001
Max. speed	2.6	4.5	3.0	<0.001	<0.001

The traditional heating cabinets show great variations in air speed from almost 0 up to 5 m/s. The flow is rotating and turbulent. The air movement is created by a fan behind a baffle, either in the back or in one side of the cabinet.

In ovens EB 01 and EB 04 the air is passed through a flowmeter with a control valve, going from the bottom to the top of the cabinet more as a laminar flow.

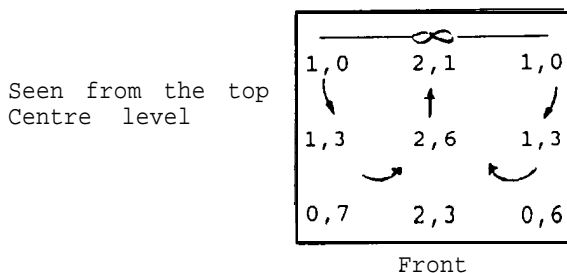


Fig. 6. Air movement and speed in UT 5042.

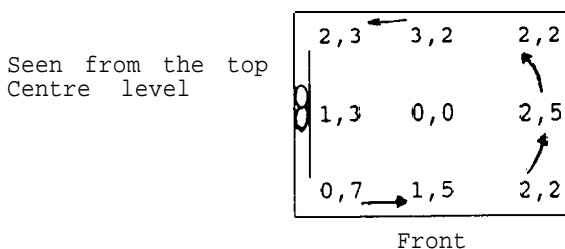


Fig. 7. Air movement and speed in UT 5060E.

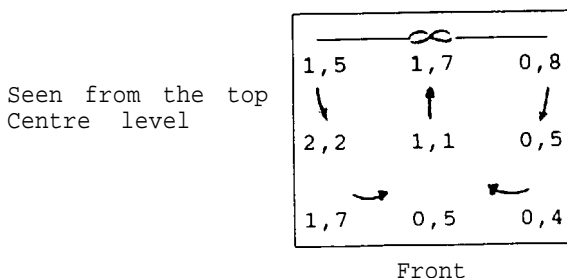


Fig. 8. Air movement and speed in TSW 60.

In the ISO 188 rubber-accelerated ageing test, no exact air speed is required, but the standard states that 'provision shall be made for a slow circulation of air through the oven of not less than three and not more than ten changes per hour'. This may be interpreted as no fans are allowed inside the testing cabinet and this means then that the three standard ovens tested may not meet the requirements for low air speed.

5.2.5 Air exchange rates

The air exchange rates have been determined both by calculating the air volume by measuring the air speed in the exhaust hole and by measuring the time needed to fill a 125 litre plastic bag attached to the exhaust hole. The results are shown in Table 6. The air exchange rate in EB 01 and EB 04 is set by a flow meter and a control valve.

TABLE 6
Air Exchange Rates

Exhaust	Air changes per hour				
	UT5042	UT 5060E	TSW60	EB 01	EB 04
Open	≈ 160	≈ 40	x 300	20	16
Closed	0	0	≈ 20	0	0

5.3 Ageing results in different ovens

To see what variations in ageing results the different ovens are causing, two kinds of ageing tests have been done, one short-term and one long-term ageing.

5.3.1 Short-term ageing

Four rubber compounds, NR, SBR, NBR and EPDM, were aged for 168 h at 100°C in four of the ovens. Before and after the ageing, microhardness and tensile tests were performed. The results are shown in Figs 9 and 10. The variations in results are calculated according to Appendix 1 and shown in Table 7 and compared with the result from a Swedish ITP in 1988 in Table 8. For formulations see Appendix 4.

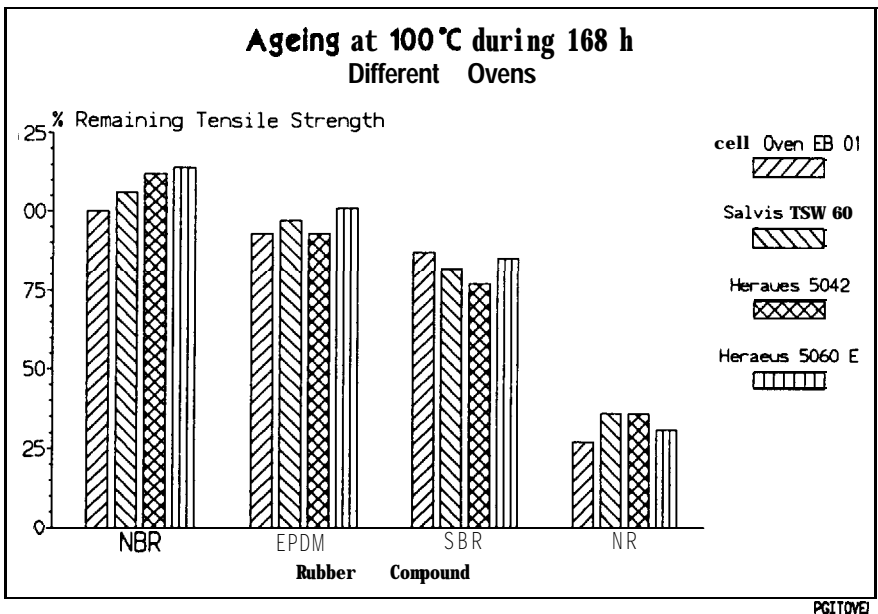


Fig. 9. Short-term ageing, tensile strength.

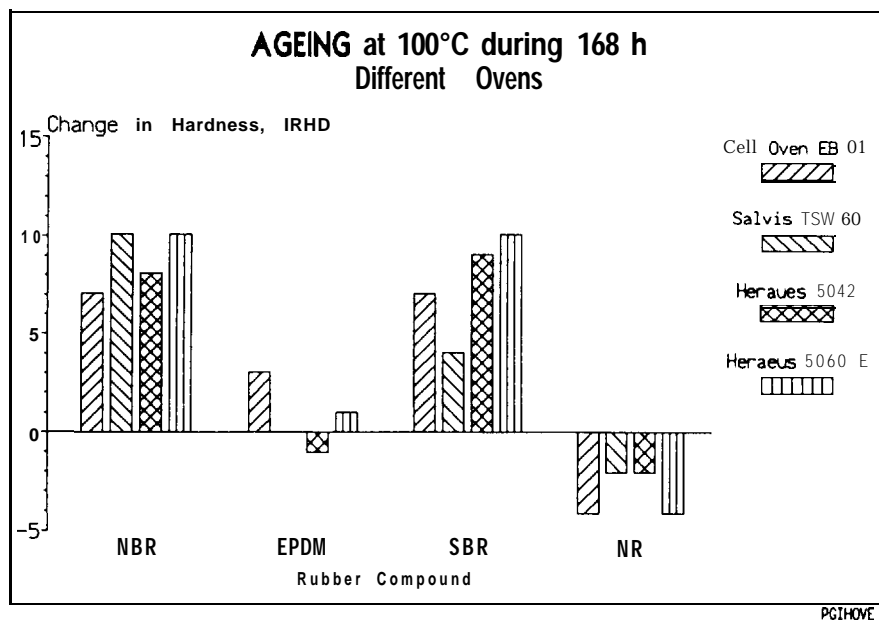


Fig. 10. Short-term ageing, hardness.

TABLE 7
Oven Influence, MicroIRHD and Tensile Test

Four different ovens	Mean	s	R
Change in tensile strength (%)	- 2 0	5	15
Change in elongation at break (%)	- 4 2	4	11
Change in microhardness, (IRHD)	3.5	1.9	5.8

TABLE 8
Ageing Test Reproduceability

12 different labs, ITP 1988	Mean	s	R
Change in tensile strength (%)	- 1 8	5.3	15
Change in elongation at break (%)	- 4 0	5.8	16
Change in microhardness, (IRHD)	- 1 3	3.8	10

s = std deviation; **R** = reproduceability in actual units of measurements.

When comparing the above results with the Swedish ITP in 1988,¹⁵ we can see that the ovens contribute most of the variations in the ageing tests. It must be noted that before the test, the temperatures of the ovens were adjusted to an actual temperature of 100°C in the centre of each oven. So most of this difference is caused by factors other than temperature.

5.3.2 Long-term ageing

One rubber compound of a NBR/PVC blend was aged in two of the ovens for 1000 h at 100°C. One Salvis TSW 60 and one Elastocon EB 01 cell oven were used. The microhardness and tensile test were performed after 72, 336 and 1000 h. The temperatures in the ovens were adjusted to be 100°C in the centre of each oven. The results are shown in Fig. 11.

The results in the figure show that the ageing doesn't follow the same course in the two ovens. The surface of the tested rubber was aged much more in the heating cabinet than in the cell oven. The main difference between the ovens was the air speed. The microhardness test is mainly measuring surface effects. The tensile test however did not show the same big differences.

The air speed seems to be of greater importance for heat ageing than is presently recognized.

5.4 Temperature influence on ageing

Four rubber compounds, NR, SBR, NBR and EPDM, were aged at three temperatures, 95, 100 and 105°C, in a cell oven for 168 h. Before and after the ageing, microhardness and tensile tests were performed. The formulations of the compounds are shown in Appendix 4.

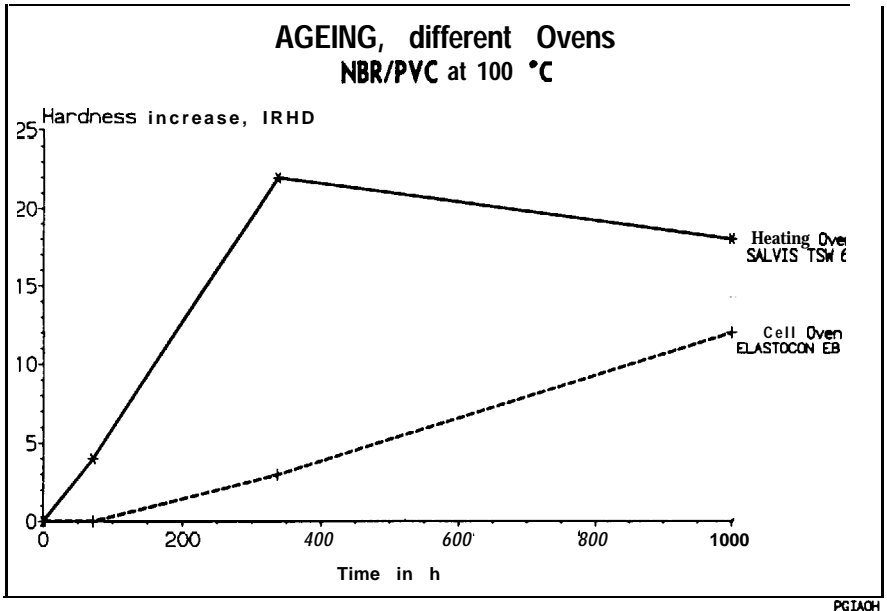
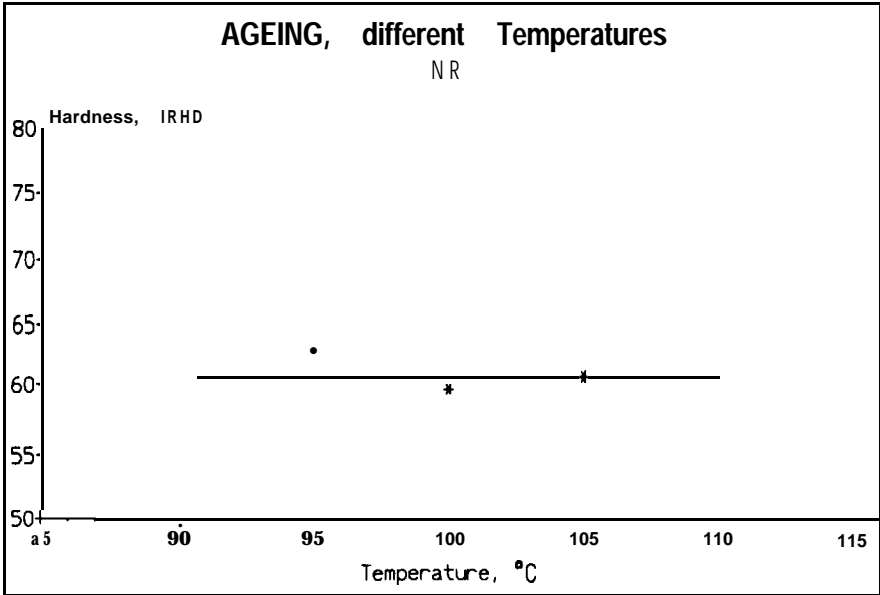


Fig. 11. Long-term ageing, micro IRHD.

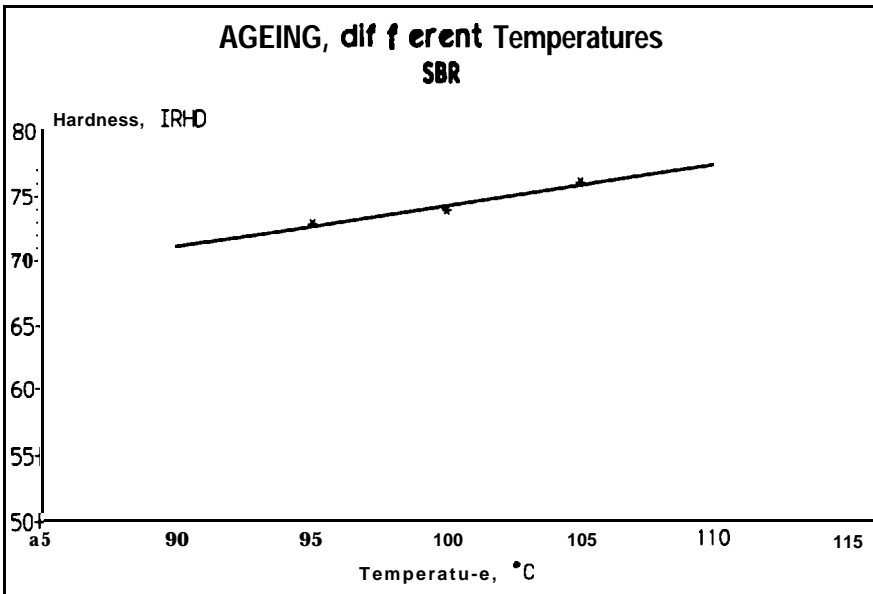
5.4.1 Change in hardness

The hardness change after 1 week of ageing at three temperatures is shown in Fig. 12-15.



PGDNR

Fig. 12. NR, change in hardness.



PGHSBR

Fig. 13. SBR, change in hardness.

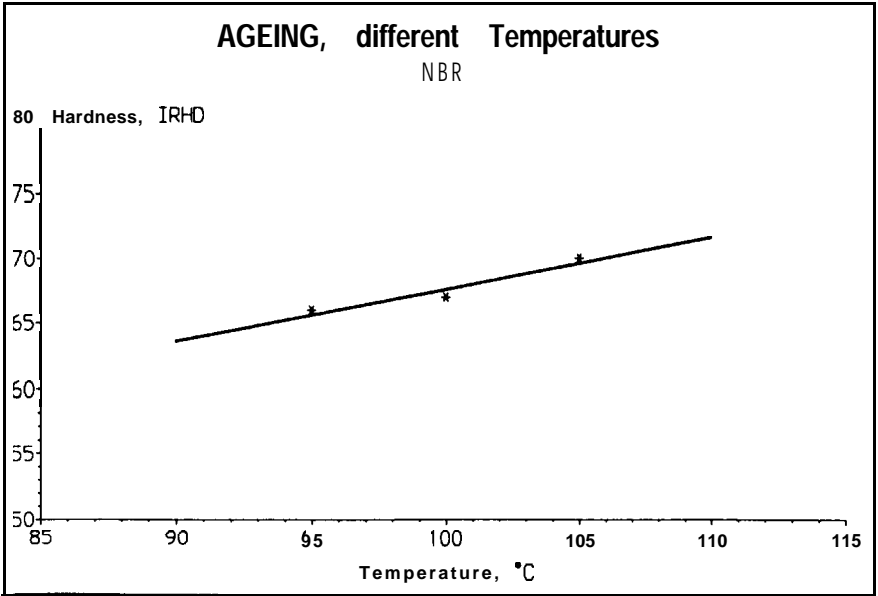


Fig. 14. NBR, change in hardness.

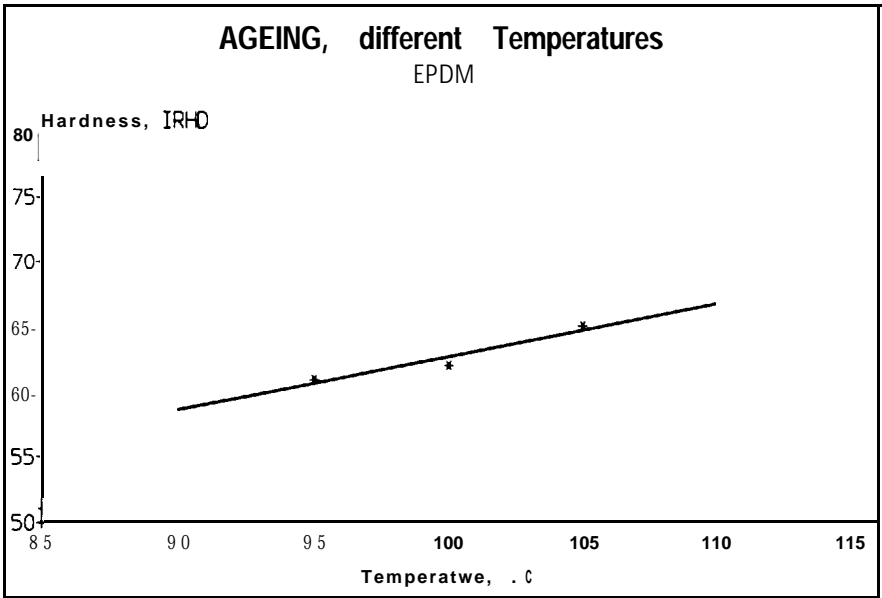


Fig. 15. EPDM, change in hardness.

5.4.2 Change in tensile properties

The change in tensile properties after 1 week of ageing at three temperatures is shown in Figs 16–19. The results show a clear influence of the temperature, even in the limited range of $\pm 5^\circ\text{C}$. The NR compound shows

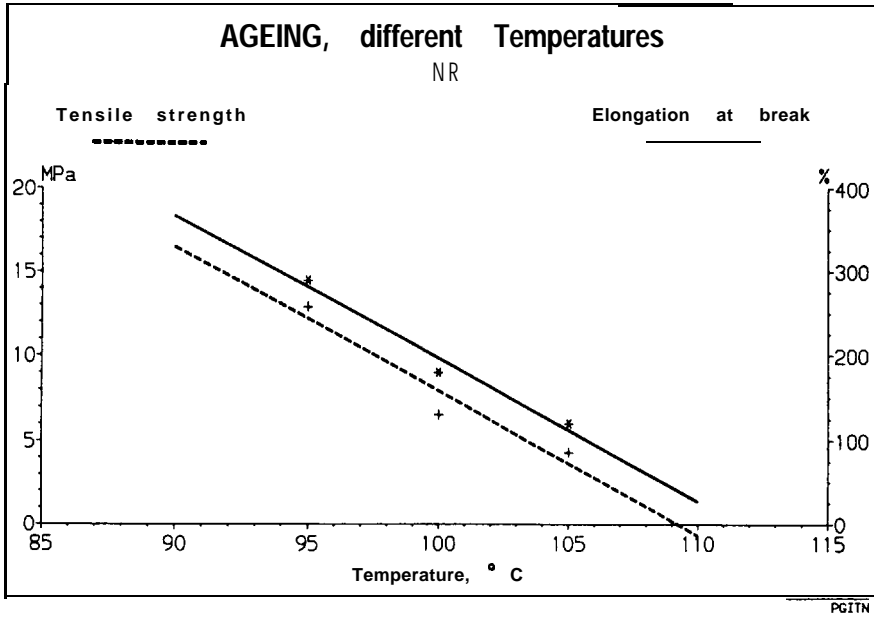


Fig. 16. NR, change in tensile properties.

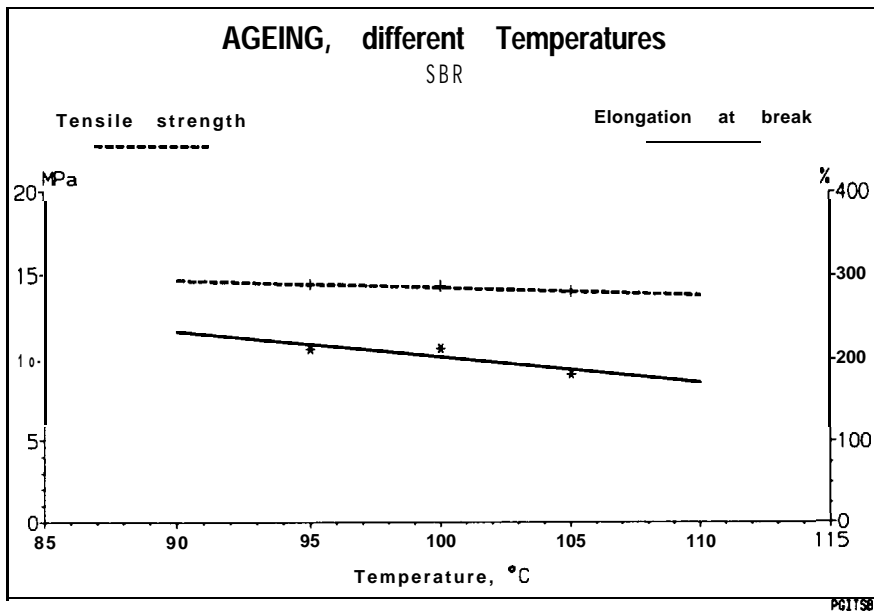
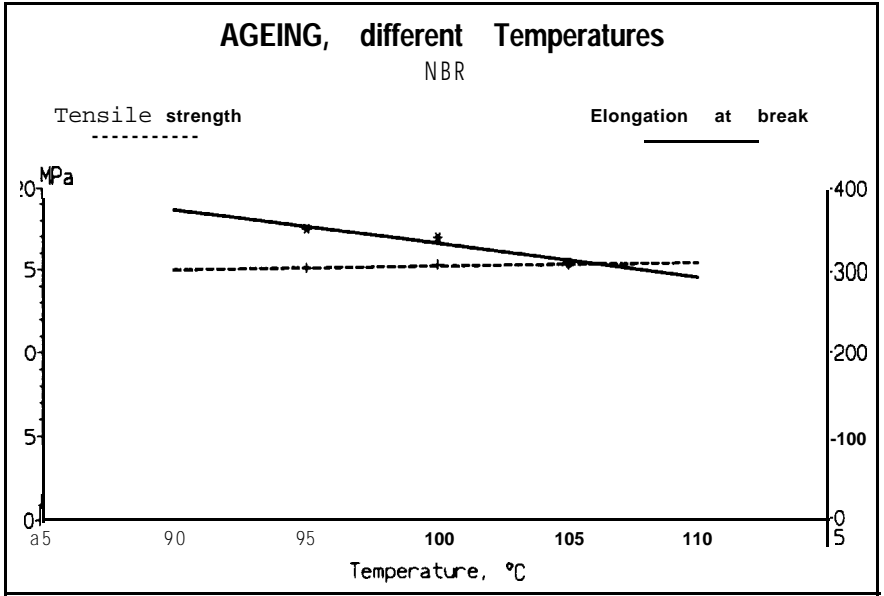


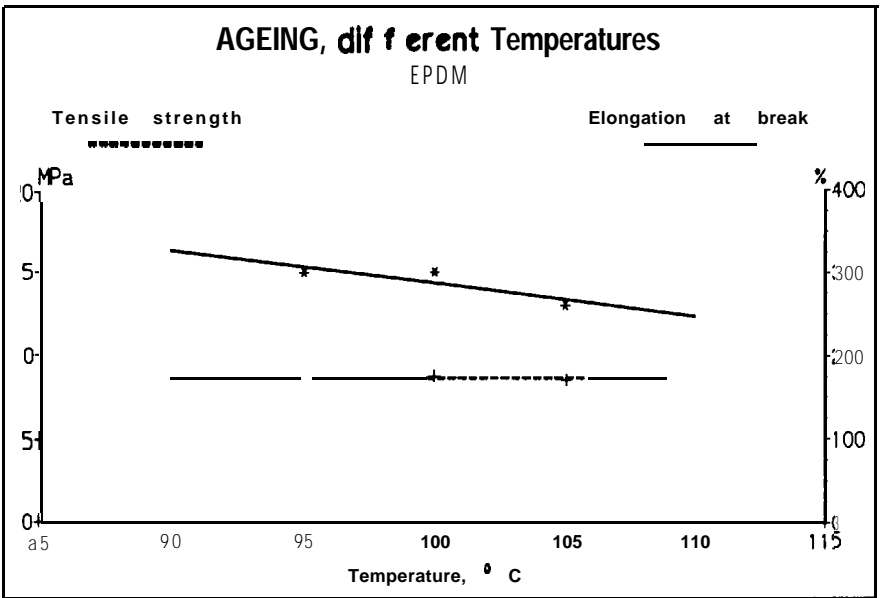
Fig. 17. SBR, change in tensile properties.

a dramatic reduction in properties in the temperature range of 100°C and the temperature seems to be too high for ageing of NR. To keep the temperature variations within close limits seems to be an important factor in reducing variations in ageing of polymers.



PG11NBR

Fig. 18. NBR, change in tensile properties.



PG11EPDM

Fig. 19. EPDM, change in tensile properties.

5.5 Air speed influence on ageing

To perform a closer investigation of the influence of air speed on heat ageing results, two special ovens were developed. One oven with an air speed of about 0.3 m/s and one with about 3 m/s. Four rubber materials, NR, SBR, NBR and EPDM, were then aged at 70°C (NR, SBR) and 100°C (NBR, EPDM) for 1000 h in these ovens plus in a cell oven with an air speed of about 0.001 m/s. Weight loss, microhardness and tensile tests were performed on the four materials after 1, 3 and 6 weeks of ageing.

5.5.1 Weight loss

The weight loss results after ageing at three air speeds are shown in Figs 20–23.

5.5.2 Hardness increase

The changes in hardness at three air speeds are shown in Fig. 2427.

5.5.3 Change in tensile properties

The change in tensile properties at three air speeds are shown in Figs 28–31. The most clear differences in properties depending on air speed were shown in weight loss and also in hardness. The other properties,

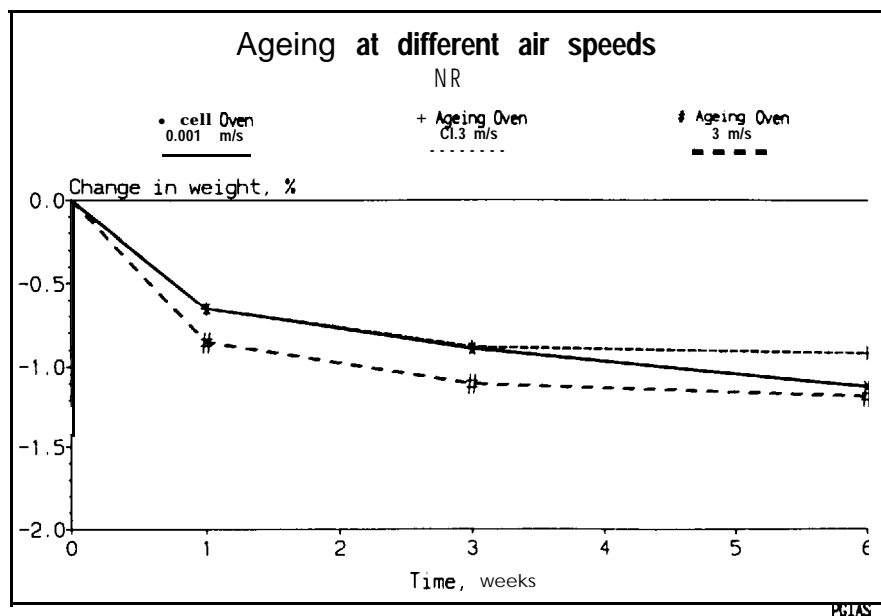


Fig. 20. NR, weight loss.

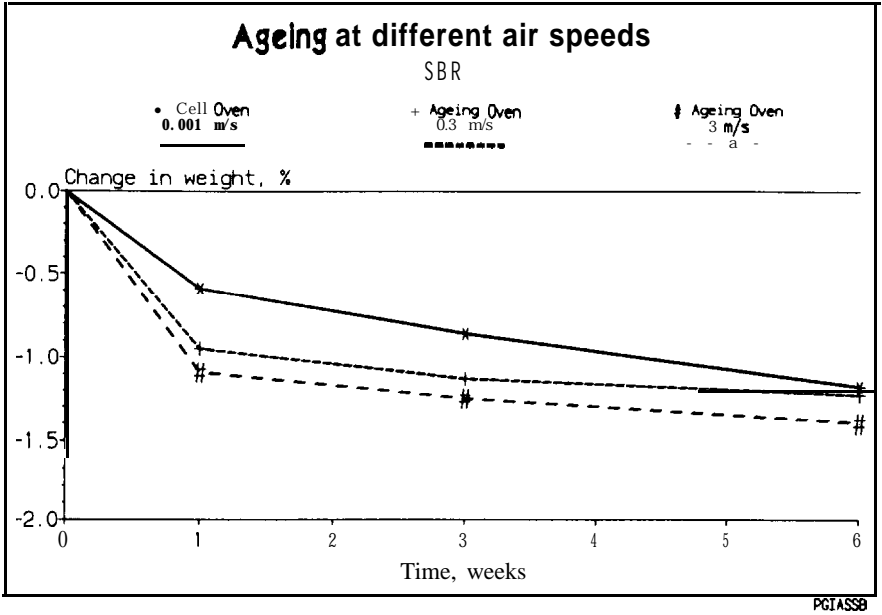


Fig. 21. SBR, weight loss.

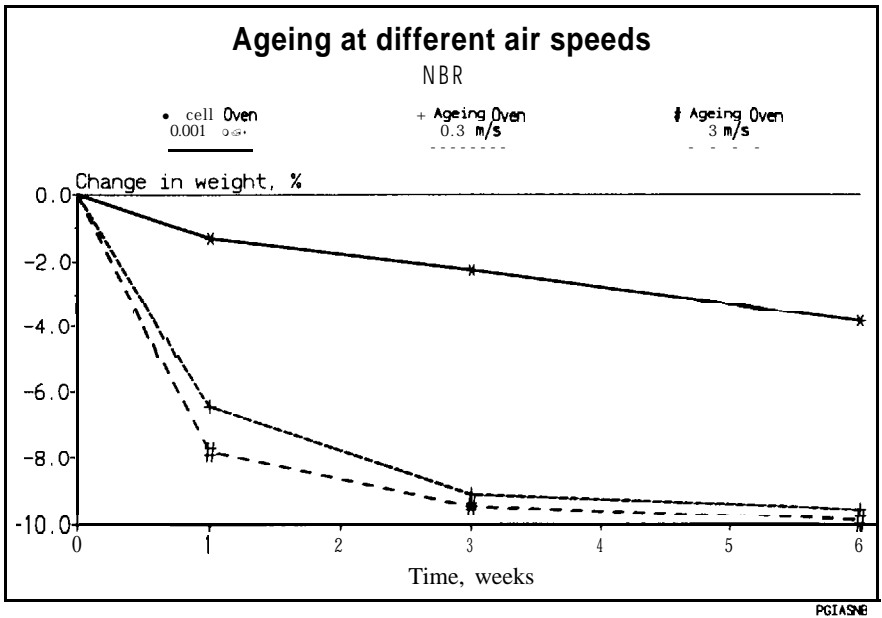


Fig. 22. NBR, weight loss.

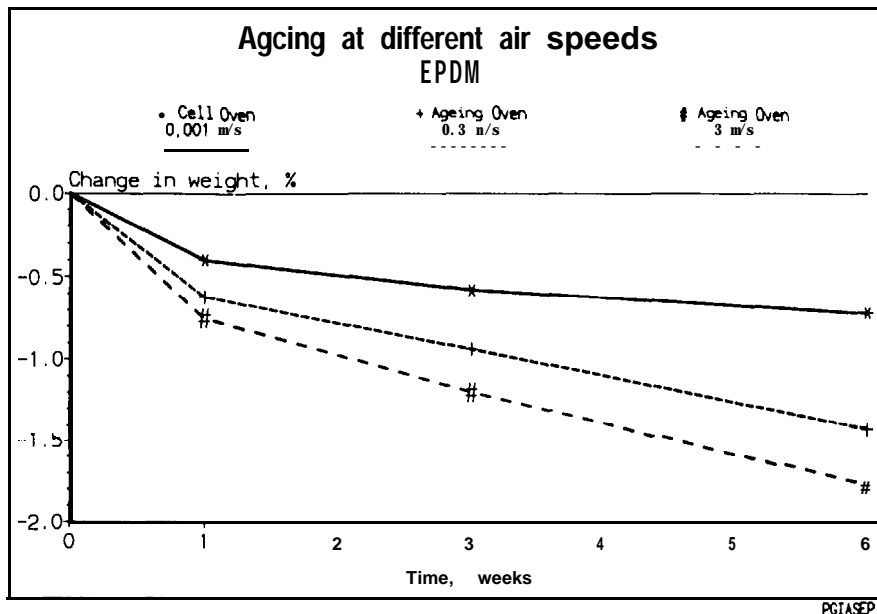


Fig. 23. EPDM, weight loss.

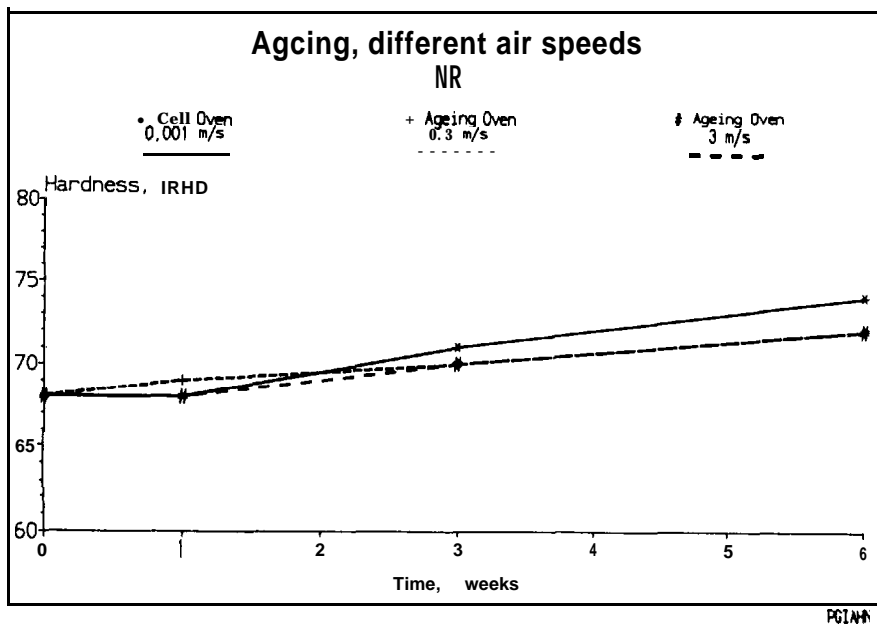


Fig. 24. NR, change in hardness.

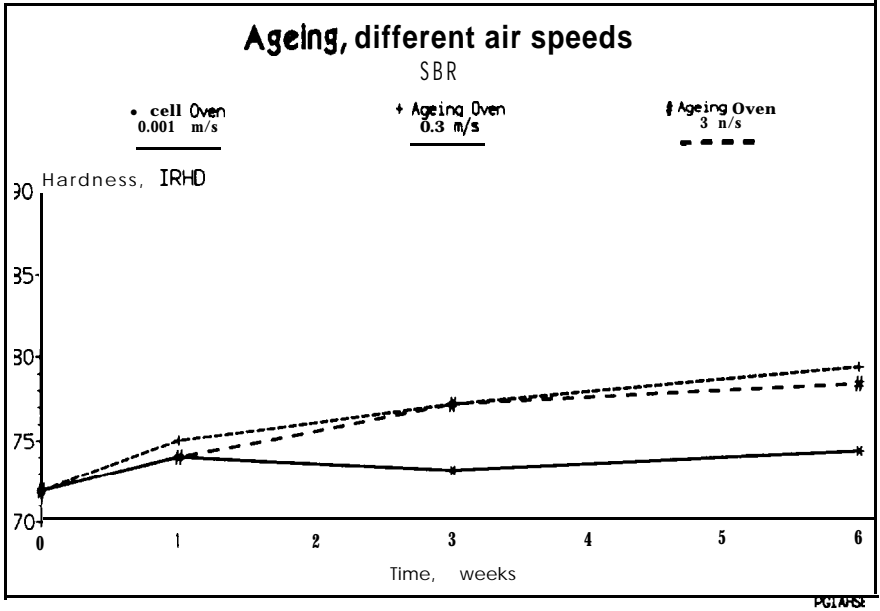


Fig. 25. SBR, change in hardness.

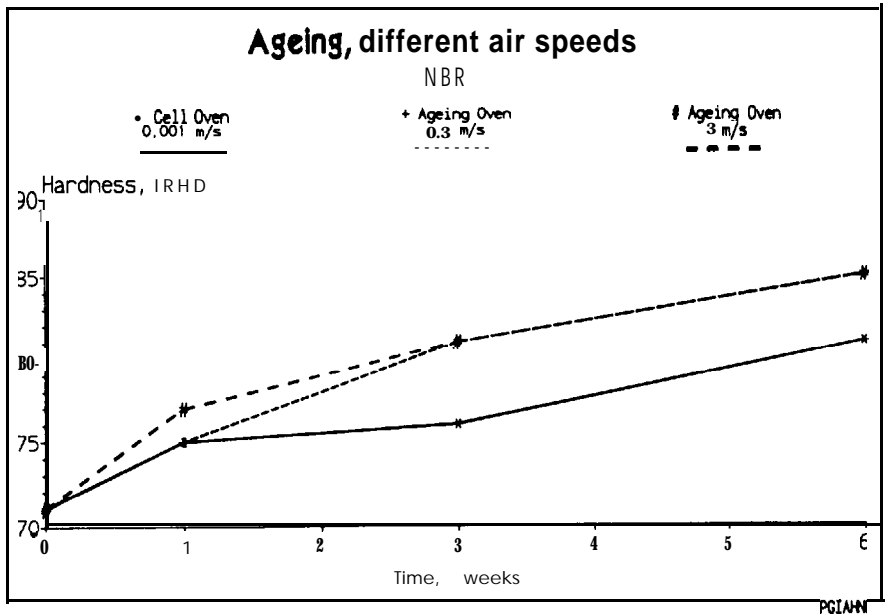


Fig. 26. NBR, change in hardness.

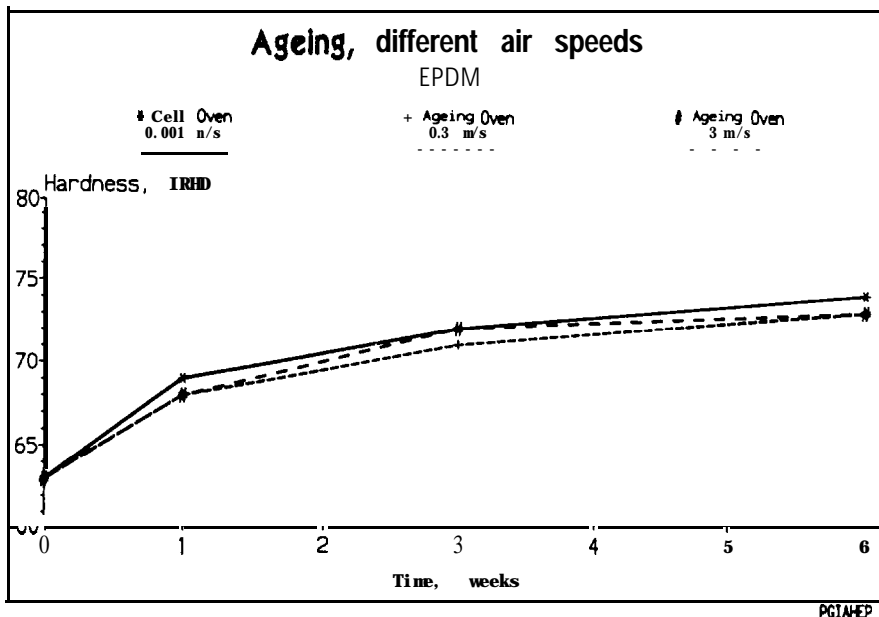


Fig. 27. EPDM, change in hardness.

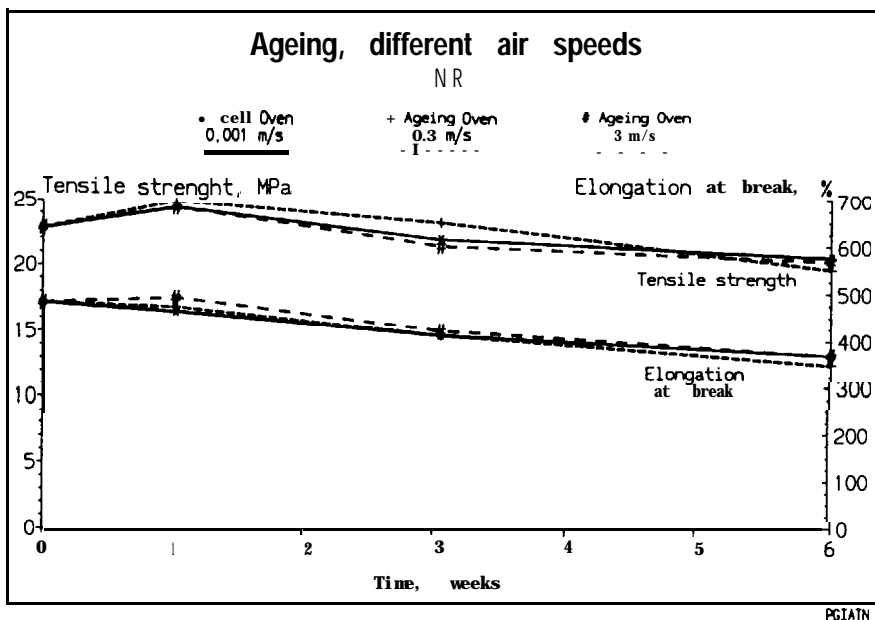


Fig. 28. NR, change in tensile properties.

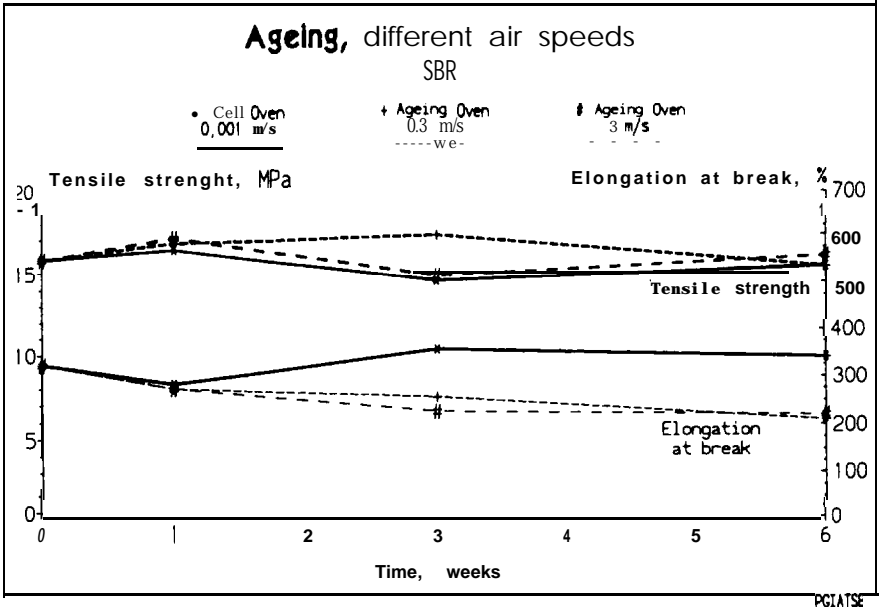


Fig. 29. SBR, change in tensile properties.

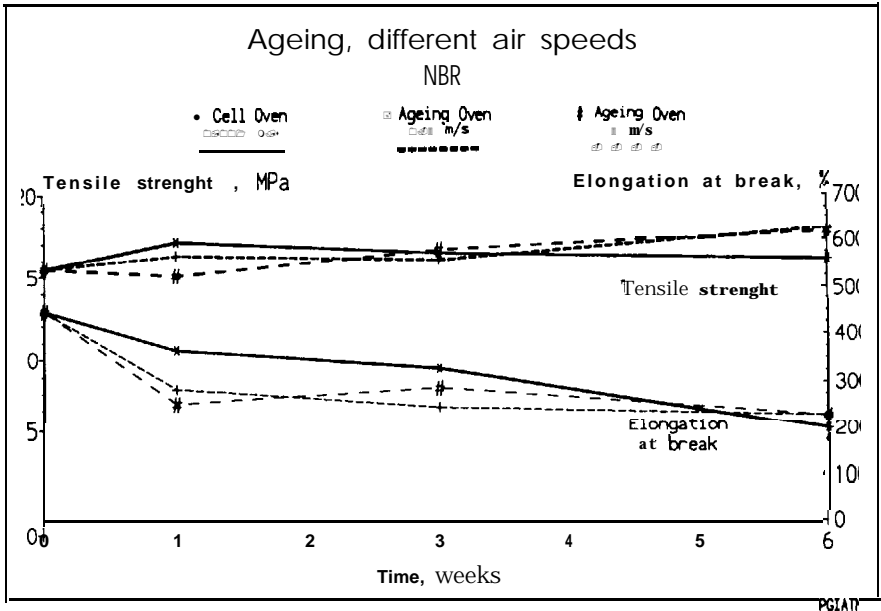


Fig. 30. NBR, change in tensile properties.

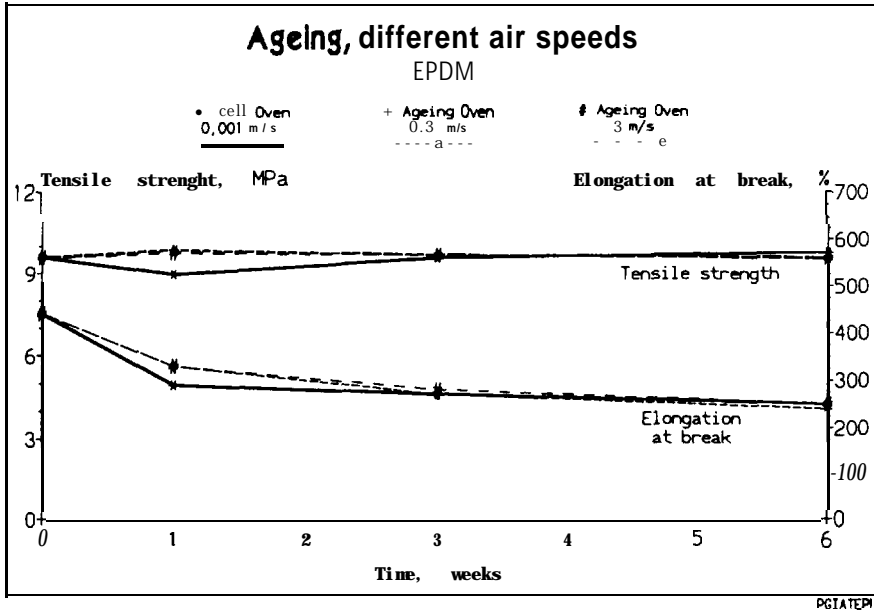


Fig. 31. EPDM, change in tensile properties.

tensile strength and elongation at break, showed differences in the test results but not any clear trends.

5.6 Air exchange rate influence on ageing

The influence of air exchange rate on ageing has not been investigated in this project as this has been done in two Interlaboratory Test Programs (ITPs) in ISO TC 45 and published in *Polymer Testing*.¹⁶ These ITPs showed only slight differences in test results between 3 and 10 air changes per hour. However at 0 air changes per hour the results showed pronounced differences. This means that it is important to have an exchange of air during ageing tests to keep the oxygen rate constant and to ventilate evaporations from the samples tested. A rate of at least 3 times per hour seems to be sufficient.

5.7 Summary of results

This project has shown that the main factors contributing to poor reproduceability between laboratories when doing heat ageing tests are air speed and temperature.

6 RECOMMENDED ACTIONS

The results from this project have shown that there is a need to improve the specifications of the ageing ovens in the standards regarding temperature and air speed. The following improvements are suggested.

6.1 Temperature

The present standard (ISO 188-82) specifies a temperature tolerance of $\pm 1^\circ\text{C}$ up to and including 100°C , $\pm 2^\circ\text{C}$ up to and including 200°C and ± 3 above 200°C .

It is suggested to reduce the tolerances in ISO 188 to 50% of present values.

It must also be clearly stated that the tolerance is valid for all variations in time and space in the test space of the ageing oven used.

It may be argued that the suggested tolerances are difficult to achieve, but it is not impossible with the technology available today.

The importance of keeping to the specified temperature during an ageing test may be illustrated with the following example: A 1°C offset during an ageing test corresponds to about 10% in test time for a material with an Arrhenius factor of 2. And we do not allow 10% variation in test time.

6.2 Air speed

ISO 188 only states that 'provision shall be made for a slow circulation of air through the oven of not less than three and not more than ten changes per hour'. This may be interpreted as meaning that no fans are allowed inside the test space of the ovens. Most laboratories seem however to use normal laboratory ovens with fans, giving problems with high air speeds.

In IEC 811 it is clearly stated that no fans are allowed inside the test space. Laboratories testing according to IEC 811 are then often using normal laboratory ovens without fans, giving problems with high temperature variations in the test space.

A reason for this situation may be that there are very few ovens specially designed for ageing tests on the market and that normal ovens are much cheaper. Laboratories then buy normal ovens as they do not realize the importance of temperature accuracy and low air speed.

It is suggested that the standard clearly defines that no fans are allowed

inside the test space and that the air *speed* shall be dependent on the air *exchange rate only*.

In addition to the present ageing test at low air speed, it is suggested to introduce a new type of heat ageing test with high air speed (1 m/s), to be able to study the influence of air speed.

An ageing test at high air speed may be of interest for materials used in products exposed to high air speeds. The air speed and direction of the air flow must however be clearly specified to achieve good reproducibility.

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4. ISO 7619-86, Rubber-determination of indentation hardness by means of pocket hardness meters.
5. ISO 37-77, Rubber, vulcanized-determination of tensile stress-strain properties.
6. ISO 188-82 Rubber, vulcanized-accelerated ageing or heat resistance tests.
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Appendix 1: HOW TO CALCULATE THE REPEATABILITY AND REPRODUCEABILITY

n = number of measured values

x_i = measurement 1, 2, 3, . . . , n

\bar{x} = mean value

$$\bar{x} = \frac{\sum x_i}{n}$$

\bar{s} = mean value (pol)

$$\bar{s} = \sqrt{\frac{\sum (s_i)^2}{n}}$$

The pol mean value is used when calculating mean values of standard deviation and coefficients of variation.

s = standard deviation

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n - 1}}$$

s_L = standard deviation between laboratories

s_r = standard deviation within laboratories

v = coefficient of variation

$$v = \frac{s}{\bar{x}} 100$$

v_L = coefficient of variation between laboratories

r = repeatability

$$r = 2.83 s_r$$

R = reproduceability

$$R = 2.83 \sqrt{s_L^2 + s_r^2}$$

If the repeatability is not calculated $s_r = 0$

Definition: An established value below which the absolute difference between two 'between - laboratory' test results may be expected to lie, with a specified probability. The probability is normally 95% if nothing else is specified.

(*R*) Reproduceability expressed as a percentage of the mean value of the measured values.

Extreme values are checked with Dixon's Outlier Test.

Appendix 2: TEST RESULTS — AGEING IN DIFFERENT OVENS AND AT DIFFERENT TEMPERATURES

Ageing, hardness (micro IRHD)

		<i>NR</i> <i>IRHD</i>	<i>SBR</i> <i>IRHD</i>	<i>NBR</i> <i>IRHD</i>	<i>EPDM</i> <i>IRHD</i>
Cell oven	O-test	64	67	60	59
	95°C	63	13	66	61
	100°C	60	74	67	62
	105°C	61	76	10	65
Salvis TSW 60	100°C	62	71	10	59
Heraeus 5042	100°C	62	16	68	58
Heraeus 5060	100°C	60	11	10	60

Ageing, tensile test

		<i>NR</i>		<i>SBR</i>		<i>NBR</i>		<i>EPDM</i>	
		<i>MPa</i>	%	<i>MPa</i>	%	<i>MPa</i>	%	<i>MPa</i>	%
Cell-oven	O-test	24.5	540	16.6	350	15.4	470	9.5	440
	95°C	12.9	290	14.5	210	15.2	350	8.6	300
	100°C	6.5	180	14.4	210	15.4	340	8.8	300
	105°C	4.3	120	14.1	180	15.4	310	8.6	260
Salvis TSW 60	100°C	8.7	200	13.6	200	16.3	330	9.2	310
Heraeus 5042	100°C	8.7	190	12.8	170	17.3	370	8.8	290
Heraeus 5060	100°C	7.7	190	14.1	180	17.6	360	9.6	310

Appendix 3: TEST RESULTS-AGEING AT DIFFERENT AIR SPEEDS

Ageing at different air speeds, 70°C: NR

		<i>Tensile strength (MPa)</i>	<i>Elongation F 100% at break (%)</i>	<i>Hardness Shore micro</i>	<i>Change in mass (%)</i>	
Cell oven	O-test	22.8	480	2.7	68	
	0.001 m/s					
	1 w	24.4	460	3.2	68	-0.65
	3 w	21.9	410	3.2	71	-0.89
	6 w	20.6	370	3.9	74	-1.12
Ageing oven	0.3 m/s					
	1 w	24.9	470	3.0	69	-0.65
	3 w	23.2	410	3.5	70	0.88
	6 w	19.7	350	4.1	72	-0.92
Ageing oven	3 m/s					
	1 w	24.4	490	2.8	68	-0.85
	3 w	21.4	420	3.1	70	-1.10
	6 w	20.3	370	3.8	72	1.18

Ageing at different air speeds, 70°C: SBR

		<i>Tensile strength (MPa)</i>	<i>Elongation F 100% at break (%)</i>	<i>Hardness Shore micro</i>	<i>Change in mass (%)</i>	
Cell oven	O-test	15.8	330	2.9	72	
	0.3 m/s					
	1 w	16.8	280	4.3	75	-0.95
	3 w	17.2	260	5.0	77	-1.13
	6 w	15.3	210	5.6	79	-1.23
Ageing oven	0.001 m/s					
	1 w	16.8	280	4.3	75	-0.95
	3 w	17.2	260	5.0	77	-1.13
	6 w	15.3	210	5.6	79	1.23
Ageing oven	3ms					
	1 w	17.1	280	3.9	74	-1.09
	3 w	14.8	230	4.5	77	-1.25
	6 w	15.9	220	5.3	78	-1.40

Ageing at different air speeds, 100°C: NBR

		<i>Tensile strength</i> (MPa)	<i>Elongation at break</i> (%)	<i>F 100%</i> (MPa)	<i>Hardness Shore micro</i>	<i>Change in mass</i> (%)
	O-test	15.5	450	2.8	71	
Cell oven	0.001 m/s					
	1 w	17.1	370	4.1	75	-1.30
	3 w	16.4	330	4.4	76	-2.24
	6 w	16.0	200	7.0	81	-3.82
Ageing oven	0.3 m/s					
	1 w	16.3	290	5.2	75	-6.46
	3 w	16.0	250	5.9	81	-9.09
	6 w	17.9	230	8.0	85	-9.54
Ageing oven	3 m/s					
	1 w	15.1	260	5.6	77	-7.80
	3 w	16.6	290	5.3	81	-9.45
	6 w	17.7	230	7.7	85	-9.89

Ageing at different air speeds, 100°C: EPDM

		<i>Tensile strength</i> (MPa)	<i>Elongation at break</i> (%)	<i>F 100%</i> (MPa)	<i>Hardness Shore micro</i>	<i>Change in mass</i> (%)
	O-test	9.6	440	2.4	63	
Cell oven	0.001 m/s					
	1 w	9.0	290	3.6	69	-0.40
	3 w	9.6	270	4.5	72	-0.58
	6 w	9.8	250	5.1	74	0.72
Ageing oven	0.3 m/s					
	1 w	9.9	330	3.3	68	-0.62
	3 w	9.7	270	4.1	71	-0.94
	6 w	9.6	240	4.3	73	-1.42
Ageing oven	3 m/s					
	1 w	9.8	330	3.5	68	-0.75
	3 w	9.7	280	4.0	72	-1.20
	6 w	9.6	250	4.2	73	-1.76

Appendix 4: FORMULATION OF RUBBER COMPOUNDS USED FOR TESTING

1. NR	<i>phr</i>
NR	100
Carbon Black N330	50
Aromatic oil	6
ZnO	5
Stearic Acid	1.5
Micro vax	2
Antioxidant, TMQ	1.0
Accelerators	2.1
Sulphur	1.6
2. SBR	
SBR	<i>100</i>
Carbon Black N330	50
Aromatic oil	6
ZnO	5
Stearic Acid	1.5
Micro vax	2
Antioxidant, TMQ	1.0
Accelerators	2.1
Sulphur	1.6
3. NBR	
NBR 33% ACN	100
Carbon Black N550	55
Plastisizer DOA	10
Activators	5
Protection	5
Accelerators	4.5
Sulphur	0.5
4. EPDM	
EPDM	100
Carbon Black N550	100
Whiting	75
Paraffin oil	100
Activators	6
Accelerators	5.75
Sulphur	1.25

Appendix: 5 THE OVENS TESTED

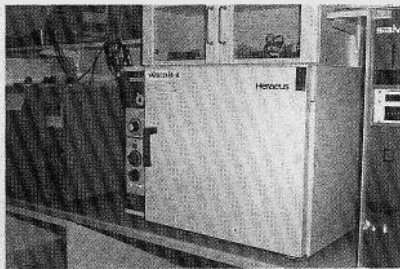
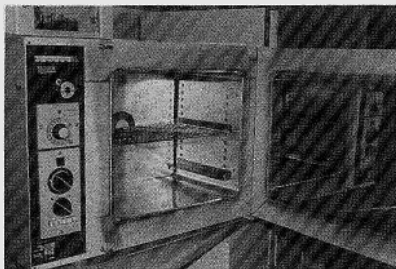


Fig. A5.1 Heraeus UT 5042.

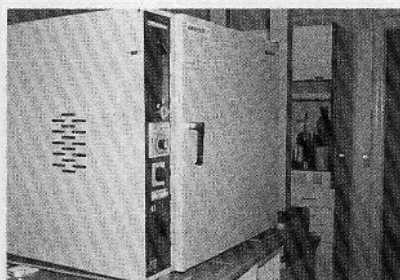
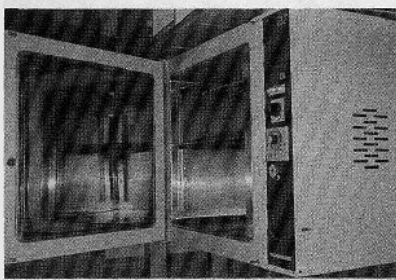


Fig. A5.2 Heraeus UT 5060E.

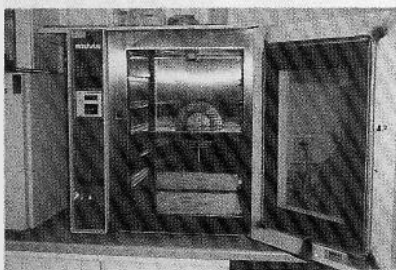


Fig. A5.3 Salvis TSW 60.

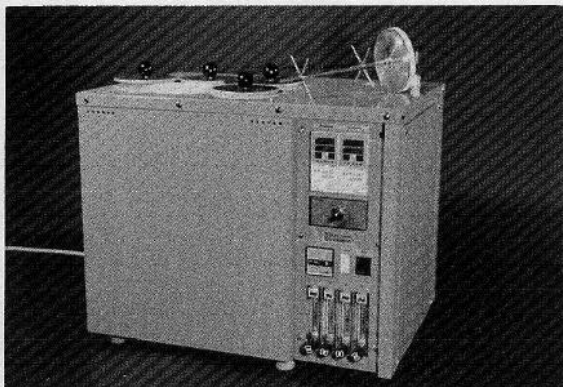


Fig. A5.4 Elastocon EB 01.

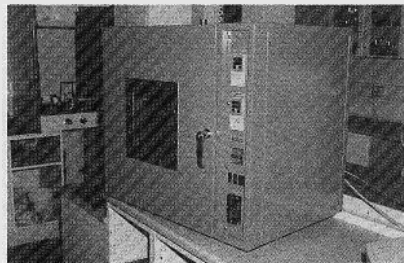
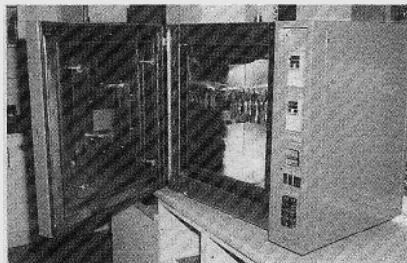


Fig. A5.5 Elastocon EB 04.