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Report

# SOME OBSERVATIONS ON THE OUTGASSING OF STAINLESS STEEL FOLLOWING DIFFERENT METHODS OF CLEANING

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1968

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SOME OBSERVATIONS ON THE OUTGASSING OF STAINLESS  
STEEL FOLLOWING DIFFERENT METHODS OF CLEANING

by

R.S. BARTON  
R.P. GOVIER

A B S T R A C T

The quantity and temperature dependence of gas released by stainless steel in vacuo following various forms of cleaning have been investigated using a high resolution mass spectrometer. The samples to be studied were introduced through a series of vacuum locks into a small oven attached to the ion source of the mass spectrometer. Each sample was heated over a period of four hours to a temperature of 450°C at which it was maintained for 16 hours. After cooling it was exposed to the air for 24 hours and the amount of gas readsorbed was investigated by an identical procedure.

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March, 1968 (BLR)

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## 1. INTRODUCTION

The work described in the following report was undertaken with a view to discovering the most satisfactory method of cleaning stainless steel vacuum vessels intended for use in plasma physics experiments in which the amount of gas evolved and its atomic number are important in determining losses from the plasma.

In order to study the total quantity and species of the gas released following different methods of cleaning, cylindrical samples of 4 cm<sup>2</sup> surface area were subjected to slow heating up to 450°C in a small oven. To reduce to a minimum discrimination of gas species caused by surface interactions resulting from collisions with walls, the oven was attached to the ion source of a high resolution mass spectrometer with the exit orifice of the oven arranged to release the gas directly into the ionization chamber.

The chosen temperature of 450°C corresponds to the limiting bake-out temperature imposed upon the majority of ultra-high vacuum equipment where borosilicate glass is used as the insulating material for electrical inputs. This temperature is also sufficiently high to allow rapid desorption of gases with activation energies between 15-25 kcal/mole which are largely responsible for prolonged surface outgassing which prevents attainment of pressures of the 10<sup>-10</sup> torr order<sup>(1,2)</sup> in unbaked systems.

Upon completion of the first outgassing cycle the samples were exposed to air with a view to simulating the re-adsorption experienced when an ultra high vacuum system is exposed to the atmosphere. The re-adsorbed gas was then examined by an identical procedure. To prevent the gas evolved from the samples being obscured by that from the walls of the system, the experiments were carried out at a pressure of below 10<sup>-9</sup> torr. Influx of air during the introduction of the sample to the oven was avoided by use of a series of vacuum locks. This allowed accurate knowledge of the outgassing properties of the oven and mass spectrometer in the absence of a sample. More complete information concerning the operation and experimental procedure is given in the subsequent sections.

## 2. APPARATUS

Observations were made using a 60° sector type mass spectrometer with a 15 cm radius of curvature and a resolution of the  $\frac{M}{\Delta m} = 130$ , for a 1% valley. Mass scanning over the range 1-300 amu in a minimum time of 1 minute was obtained using a variable magnetic field and fixed accelerating voltage. A partial pressure of 10<sup>-12</sup> torr was observable using an electron multiplier as a means of ion current amplification.

The danger of hydrocarbon contamination of the experimental chamber resulting from the pumping system was avoided by use of mercury diffusion pumps. Continuous operation of the apparatus over several weeks was facilitated by the use of a drip-back chevron baffle below the nitrogen trap. As a further precaution against hydrocarbon contamination the inner lock, experimental chamber and analyser tube pumps were exhausted into a ballast tank via a mercury diffusion pump with a critical backing pressure of about 30 torr (4 stage). This avoided the continuous use of a rotary backing pump.

The apparatus was built of 18/8/1 stainless steel and with the exception of the outer vacuum lock was bakeable to 450°C. All demountable joints in the bakeable zone had gold wire seals. Indium was used in the unbaked region. The machine was divided into three stages - an outer vacuum lock, an inner vacuum lock and an experimental chamber. The outer lock A (Fig.1) was used as a means of introducing the sample to the vacuum system, B,C, without raising it to atmospheric pressure. It also served as the body of a metal seated valve, D, which separated it from the inner lock, B. The shaft of the valve contained a cylindrical cavity cut obliquely in it radially to form a drawer housing the sample which was inserted into it through the port, E, following removal of the sealing plate.

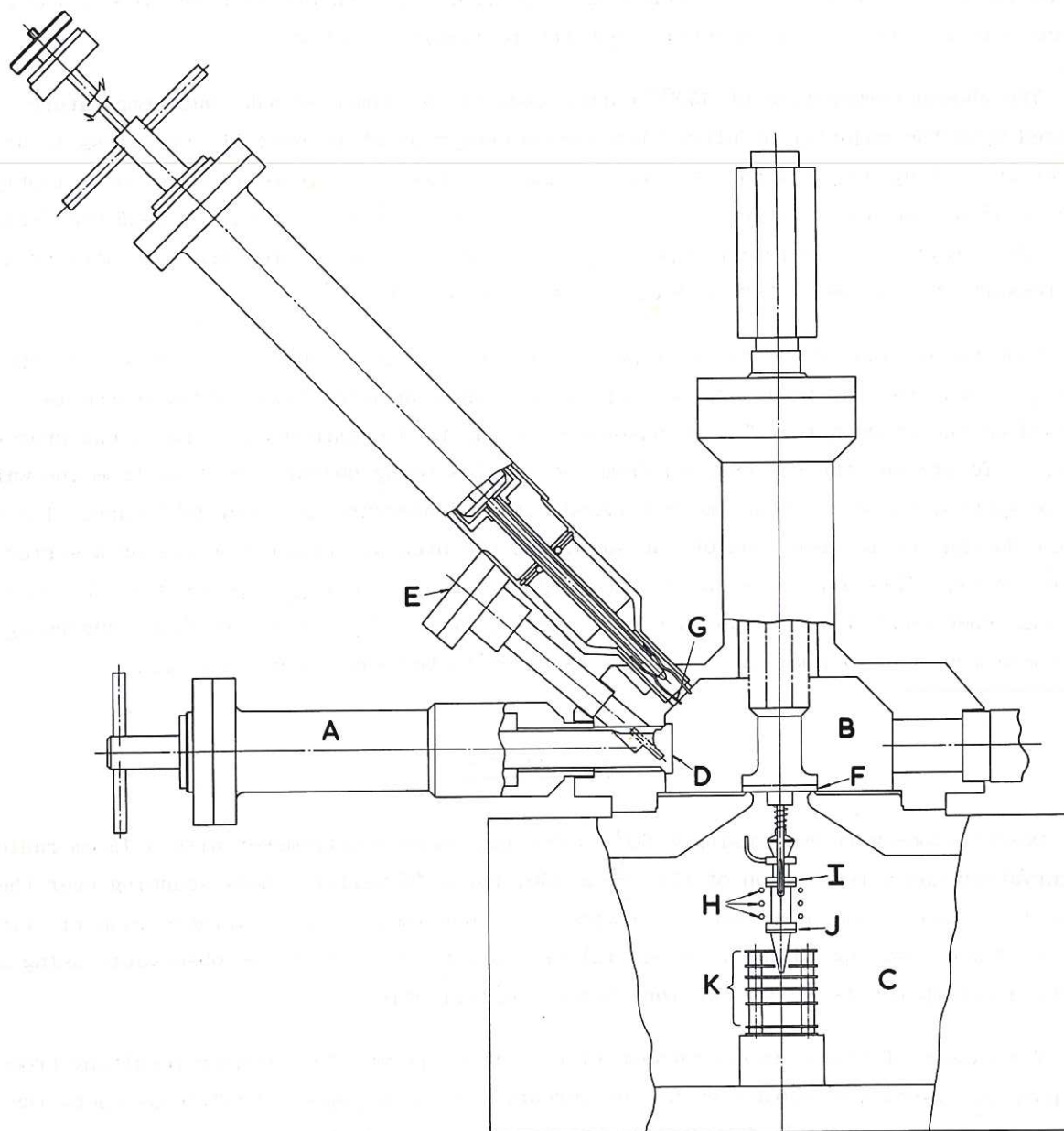


Fig.1 Sample loading assembly and ion source (CLM-R 93)

The inner lock, B, was an intermediate zone of communication between the outer lock and the experimental chamber and was pumped to a base pressure of  $10^{-9}$  torr. Its presence prevented the metal seated valve, F, from experiencing an atmosphere pressure gradient during the introduction of the sample. Transfer of the sample from the outer to the inner lock was facilitated by opening the valve, D, which propelled it into the correct position to be picked up by the tongs, G, which were operated manually by a system of metal flexible bellows.

The experimental chamber contained the mass spectrometer ion source, analyser tube and a small oven, I, in which the sample was placed for examination. The oven was suspended from the base of the seating plunger of the valve, F. Closure of the valve incorporated automatic manipulation of the oven causing it to come to rest on a ground seating ring, J, and at the same time closing the spring loaded lid. This ensured that both lid and oven were held firmly in position. Opening the valve also opened and lifted the oven to the entrance of the inner lock tilting it into the correct position to receive or give up the sample. To ensure that the maximum quantity of gas from the sample entered the ionization chamber the ground seating ring supporting the oven was mounted on the top of the ion source, K, with its gas outlet entering the chamber.

The temperature of the oven was raised to the maximum required ( $450^{\circ}\text{C}$ ) by radiation from a tungsten filament, H, surrounding it. To prevent the outer walls of the experimental chamber giving increased outgassing due to temperature rise in the presence of radiation from the heater filament, they were water cooled.

The analyser tube was differentially pumped to avoid gas produced by ion bombardment of the walls influencing the gas composition in the experimental chamber. These both had a base pressure of about  $5 \times 10^{-10}$  torr.

### 3. EXPERIMENTAL PROCEDURE

A cylindrical sample of  $4 \text{ cm}^2$  surface area was placed in the outer vacuum lock A (Fig.1) where it was pumped for 10 minutes at the end of which time the pressure was sufficiently low to allow it to be transferred to the inner lock where it was removed from the conveyor by the tongs, G. The inner and outer locks were then isolated from each other and pumping of the inner lock and sample was allowed to continue for a further five minutes. This time interval was the minimum required to eliminate the effects of gas influx from the outer lock in the absence of a sample. It was also chosen as a convenient time in which to make a mass spectrum of the residual gas composition.

At the end of this period the sample was inserted into the oven, I. After a further five minutes the temperature of the oven was raised over a period of four hours to  $450^{\circ}$  at which it was maintained for sixteen hours. Mass spectra were made at frequent intervals to obtain detailed information regarding the quantity and composition of the gas evolved throughout the entire experimental period. A continuous chart recording showing time and temperature relationship was also made.

An identical series of observations was carried out without the sample present during

the twenty-four hours immediately preceding sample injection, the two being separated by a four hour cooling period. The partial pressures of the gas components measured at intervals during this heat cycle were subtracted from those obtained at identical temperatures with the sample present enabling the quantity and composition of the gas evolved by the sample to be related to time and temperature. Following the initial baking cycle and re-exposure of the sample to atmosphere for twenty-four hours, the cycles were repeated in the same order to determine the quantity of gas re-adsorbed.

#### 4. PROCESSES EXAMINED

The following processes were examined:-

1. Dry Machining.
2. Machining using cutting fluid.
3. Coarse machining using cutting fluid.
4. Electropolishing.
5. Vapour blasting.
6. Air baking at 450°C.

##### Vapour degreasing

In order to simulate the conditions prevailing during assembly of a large equipment, each of the processes examined was followed by vapour degreasing. This was carried out in a vapour bath consisting of hot "Triklone B" (stabilized trichloroethylene).

##### Electropolishing

The bath used for this process was of the following composition:-

|                                                                          |                     |
|--------------------------------------------------------------------------|---------------------|
| Orthophosphoric acid S.G.1.75,                                           | 350 parts by volume |
| N. butyl alcohol,                                                        | 90 parts by volume  |
| Temp. of use                                                             | 70°C                |
| Amount of material removed from surface of sample $3 \times 10^{-3}$ cm. |                     |

Electropolishing was followed by thorough washing in distilled water.

##### Vapour blasting

The specimen was cleaned by blasting with glass powder of particle size  $1 - 1.5 \times 10^{-2}$  mm (Vaquashene 10/15 medium)\* suspended in water containing a degreasing agent (Vaqua 'D' chemical)\*. This was applied at a nozzle pressure of 100 psi. The treatment was followed by thorough washing in distilled water.

##### Air baking

The sample was baked in air for twenty-four hours at 450°C. This process has been recommended by Petermann<sup>(3)</sup> for reducing outgassing. The improvement is thought to be due to the thin layer of oxide forming a barrier to diffusion of gas, particularly of hydrogen, from the bulk of the material material to the surface.

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\* Supplied by Abrasive Developments Ltd.



## 5. DISCUSSION OF RESULTS

### Factors affecting outgassing

Cutting oil is normally used during the machining of stainless steel to assist in the production of a high quality finish. The effect of this on the outgassing rate of the material was studied by comparing a sample machined using the cutting oil currently in use (Duckham's Aquacut 40) (sample 2) with a dry machined sample (sample 1). The resultant outgassing (Table 1) was in favour of machining using the lubricant, and for this reason cutting oil was used in the machining of all subsequent samples.

During the first outgassing cycle when the gas content of the specimen depended largely on the conditions of manufacture the electropolished sample (4) showed the lowest outgassing, followed by the vapour blasted (5) and airbaked (6) specimens (Table 1), the order depending on the efficiency in removing dirt. When the gas content resulting from the previous history had been removed by the initial baking cycle, however, the arrangement changed and during the bake following exposure to air outgassing rates corresponded (with the exception of the air baked specimen) to surface finish.

Assuming, therefore, that the outgassing during the second bake is proportional to the surface area (see also discussion of page 13) then the ratio of outgassing during the first cycle to that during the second is a measure of the cleaning efficiency. On this basis vapour blasting and air baking seem to be the best cleaning techniques. However, since they both produce high specific surface areas they are not the best techniques for practical purposes.

Examination of the three methods which removed the original dirt most satisfactorily showed that two of these removed the surface layer and the third probably removes the dirt by oxidation and evaporation. Electropolishing, which removes the surface material and leaves a high polish showed the lowest practical outgassing rate during both cycles, but it was only marginally better during the second cycle than the sample on which lubrication was used during machining. The high outgassing rate exhibited by the air baked sample during the second cycle suggests an unusually high specific surface area. In view of the exceptional results obtained from the specimen further study of the technique is being made.

### Total gas released

The total gas released during the first heat cycle varied from approximately  $2 - 14 \times 10^{-3}$  torr litres  $\text{cm}^{-2}$  (Table 1). Following the initial degassing and re-exposure to atmosphere the total gas released during the subsequent heat cycle was generally at least one order of magnitude less. More detailed information regarding the variations in the rate of evolution of the major outgassing components from each sample with time and temperature is shown in Figs. 2 - 13.

TABLE 1

Total gas evolved during bake at 450°C, based on the ion source sensitivity for N<sub>2</sub>  
 (Samples arranged in order of surface finish)

| Sample          |     | Surface finish<br>μ inches | Total gas evolved            |                          |                              |                          | Ratio<br>1st Bake<br>2nd Bake |
|-----------------|-----|----------------------------|------------------------------|--------------------------|------------------------------|--------------------------|-------------------------------|
|                 |     |                            | 1st Bake                     |                          | 2nd Bake                     |                          |                               |
| Process         | No. |                            | Torr litres cm <sup>-2</sup> | Equivalent<br>Monolayers | Torr litres cm <sup>-2</sup> | Equivalent<br>Monolayers |                               |
| Electropolished | 4   | 14                         | 247 x 10 <sup>-5</sup>       | 82                       | 21 x 10 <sup>-5</sup>        | 7                        | 12                            |
| Using lubricant | 2   | 31                         | 345 x 10 <sup>-5</sup>       | 114                      | 32 x 10 <sup>-5</sup>        | 11                       | 10                            |
| Vapour blasted  | 5   | 36                         | 290 x 10 <sup>-5</sup>       | 96                       | 60 x 10 <sup>-5</sup>        | 20                       | 5                             |
| Dry machined    | 1   | 46                         | 960 x 10 <sup>-5</sup>       | 316                      | 68 x 10 <sup>-5</sup>        | 22                       | 14                            |
| Air baked       | 6   | 78                         | 310 x 10 <sup>-5</sup>       | 102                      | 164 x 10 <sup>-5</sup>       | 54                       | 2                             |
| Coarse machined | 3   | 160                        | 1400 x 10 <sup>-5</sup>      | 460                      | 80 x 10 <sup>-5</sup>        | 26                       | 18                            |

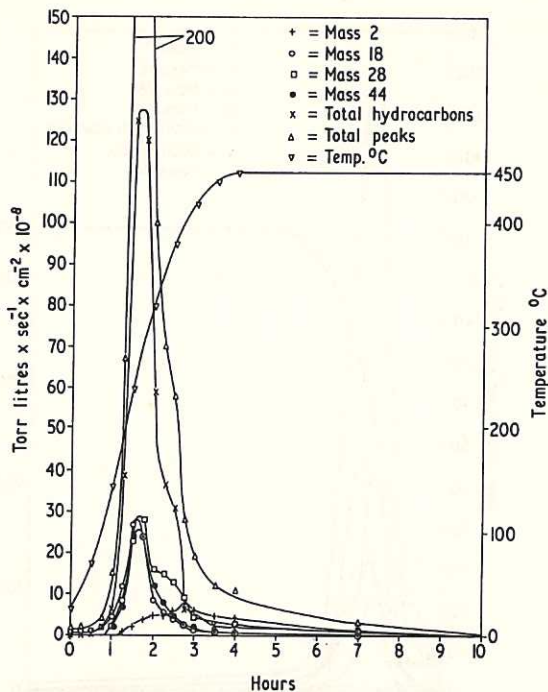


Fig. 2 (CLM-R 93)  
 Outgassing during 1st bake-out cycle of dry machined sample. Matl. 18/8/1 Expt. 29. Sample 1. Date 13-15/12/66. Max. temp. 450°C. Surface finish 46 μ in

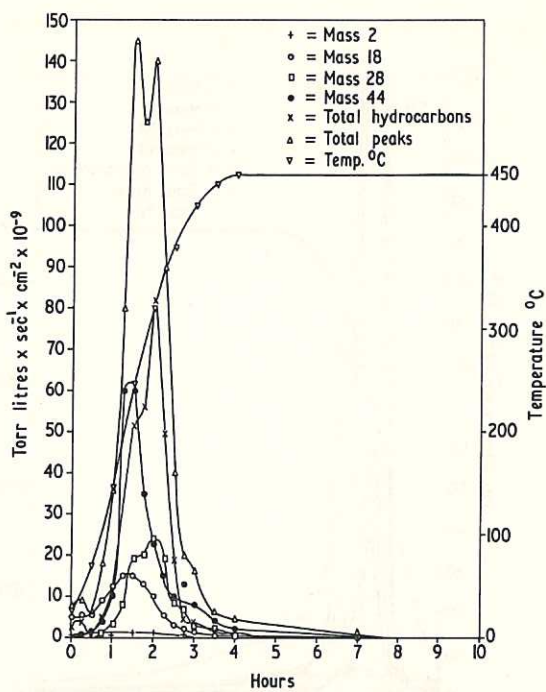


Fig. 3 (CLM-R 93)  
 Outgassing during 2nd bake-out cycle (following exposure to atmos.) of dry machined sample. Matl. 18/8/1. Expt. 30, sample 1. Date 15-17/12/66. Max. temp. 450°C. Surface finish 46 μ in

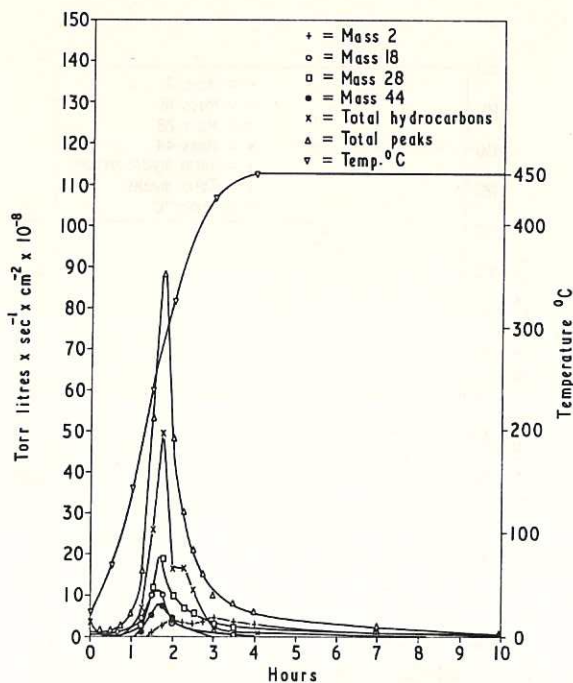


Fig. 4 (CLM-R 93)  
 Outgassing during 1st bake-out cycle of sample machined using cutting oil. Matl. 18/8/1. Expt. 25. Sample 2. Date 29/11/66. Max. temp. 450°C. Surface finish 31 μ in

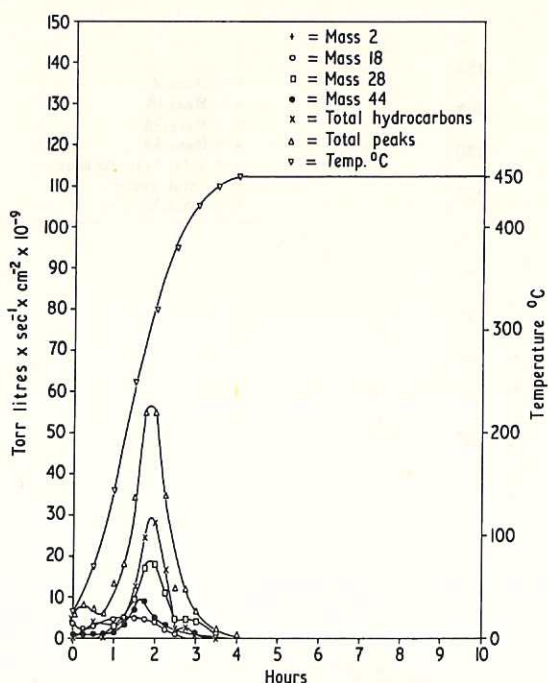


Fig. 5 (CLM-R 93)  
 Outgassing during 2nd bake-out cycle (following exposure to atmos.) of sample machined using cutting oil. Matl. 18/8/1 Expt. 26. Sample 2. Date 1-3/12/66. Max temp. 450°C. Surface finish 31 μ in

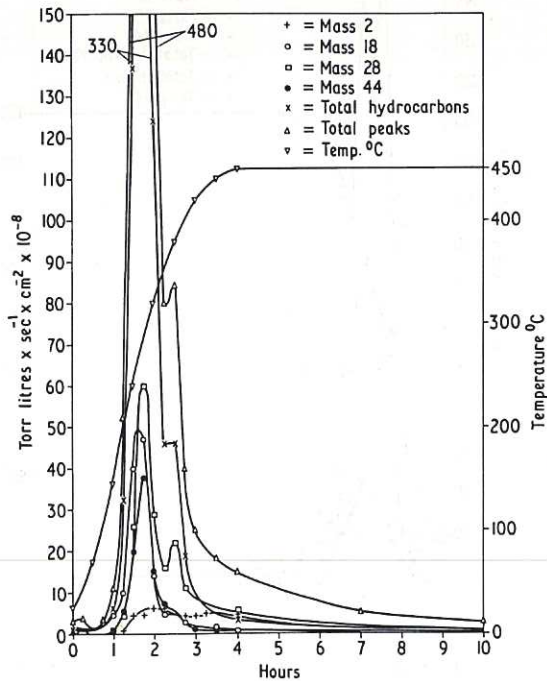


Fig. 6 (CLM-R 93)  
 Outgassing during 1st bake-out cycle of coarse machined sample. Matl. 18/8/1. Expt. 27. Sample 3. Date 6-8/12/66 Max temp. 450°C. Surface finish 160  $\mu$  in

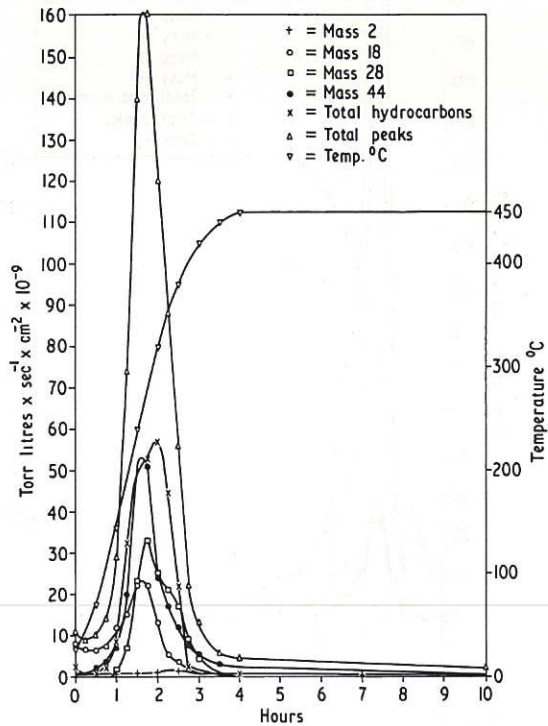


Fig. 7 (CLM-R 93)  
 Outgassing during 2nd bake-out cycle (following exposure to atmos.) of coarse machined sample. Matl. 18/8/1. Expt. 28. Sample 3. Date 8-9/12/66. Max. temp. 450°C. Surface finish 160  $\mu$  in

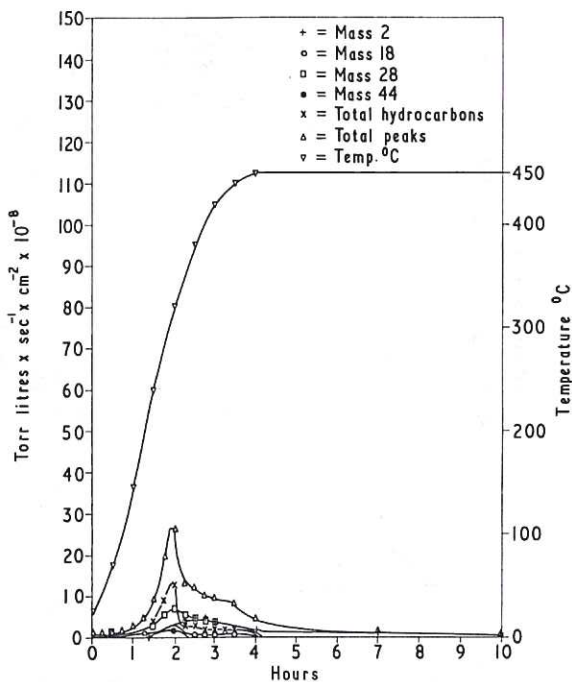


Fig. 8 (CLM-R 93)  
 Outgassing during 1st bake-out cycle of electro polished sample. Matl. 18/8/1. Expt. 23. Sample 4. Date 22-23/11/66. Max. temp. 450°C. Surface finish 14  $\mu$  in

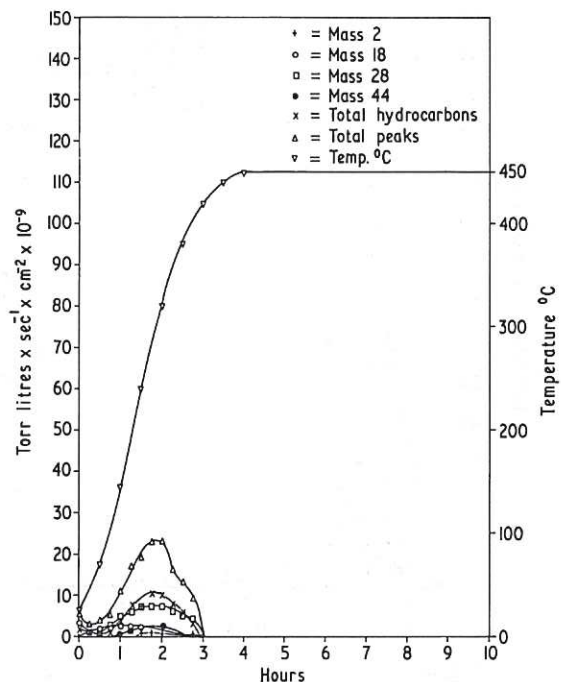


Fig. 9 (CLM-R 93)  
 Outgassing during 2nd bake-out cycle (following exposure to atmos) of electro polished sample. Matl. 18/8/1. Expt. 24. Sample 4. Date 24/11/66. Max. temp. 450°C. Surface finish 14  $\mu$  in

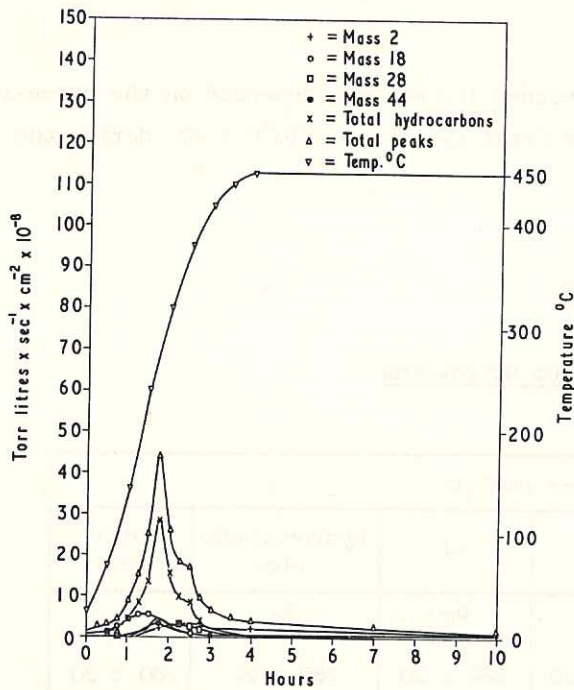


Fig. 10 (CLM-R 93)  
 Outgassing during 1st bake-out cycle of vapour blasted sample. Matl. 18/8/1. Expt. 31. Sample 5. Date 11-13/1/67. Max. Temp. 450°C. Surface finish 36  $\mu$ in

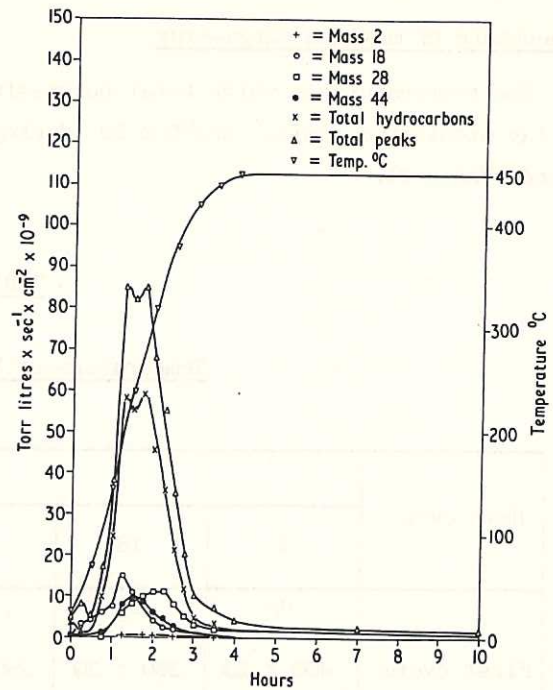


Fig. 11 (CLM-R 93)  
 Outgassing during 2nd bake-out cycle (following exposure to atmos.) of vapour blasted sample. Matl. 18/8/1. Expt. 32. Sample 5. Date 13-15/1/67. Max. temp. 450°C. Surface finish 36  $\mu$ in

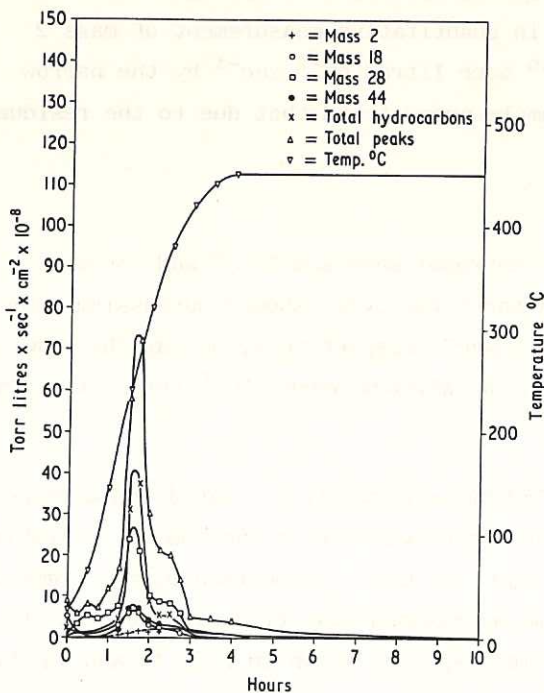


Fig. 12 (CLM-R 93)  
 Outgassing during 1st bake-out of air baked sample. Matl. 18/8/1. Expt. 16. Sample 6. Date 6/9/67. Max. temp. 450°C. Surface finish 78  $\mu$ in

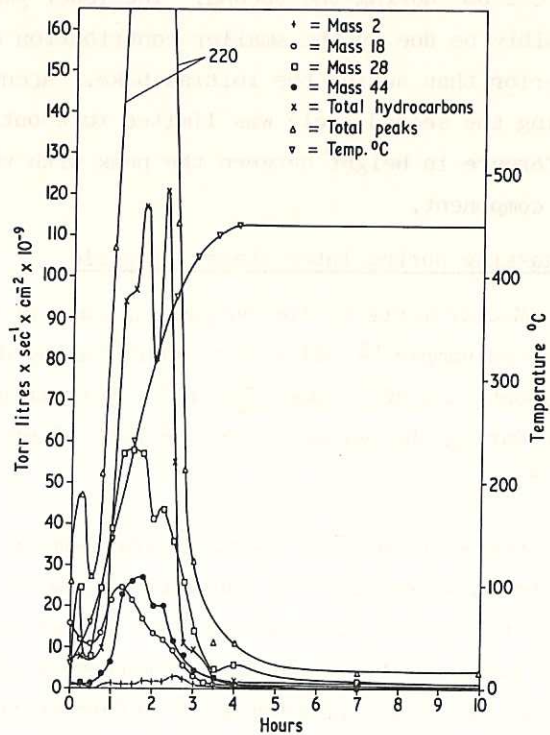


Fig. 13 (CLM-R 93)  
 Outgassing during 2nd bake-out cycle (following exposure to atmos.) of air baked sample. Matl. 18/8/1. Expt. 17. Sample 6. Date 8/9/67. Max. temp. 450°C. Surface finish 78  $\mu$ in

### Temperature of maximum outgassing

The temperature at which total outgassing reached its maximum depended on the process, but lay within the limits  $280^{\circ}\text{C} \pm 20$  during the first cycle and  $270^{\circ}\text{C} \pm 40$  during the second (Table 2).

TABLE 2

### Temperature of Maximum Outgassing

| Heat cycle   | Mass number        |                    |                    |                    |                    |                    |
|--------------|--------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
|              | 2                  | 18                 | 28                 | 44                 | Hydrocarbons etc.  | Total gas          |
|              | $^{\circ}\text{C}$ | $^{\circ}\text{C}$ | $^{\circ}\text{C}$ | $^{\circ}\text{C}$ | $^{\circ}\text{C}$ | $^{\circ}\text{C}$ |
| First cycle  | $400 \pm 20$       | $280 \pm 20$       | $280 \pm 20$       | $280 \pm 20$       | $280 \pm 20$       | $280 \pm 20$       |
| Second cycle | $330 \pm 50$       | $210 \pm 20$       | $280 \pm 40$       | $260 \pm 20$       | $270 \pm 40^*$     | $270 \pm 40^*$     |

\* Excluding air baked sample.

Hydrogen reached its peak of outgassing at  $400^{\circ}\text{C} \pm 20$  during the first cycle and  $330^{\circ}\text{C} \pm 50$  during the second. The lower temperature shown during the second cycle may possibly be due to the smaller contribution made by the diffusion of hydrogen from the interior than during the initial bake. Accuracy in quantitative measurement of mass 2 during the second cycle was limited to about  $10^{-10}$  torr litres  $\text{cm}^{-2} \text{sec}^{-1}$  by the narrow difference in height between the peak with the sample present and that due to the residual gas component.

### Outgassing during later stages of cycle

Measurements of the outgassing rate of the electropolished sample (4) and the well machined sample (2) after five hours baking during the first cycle showed outgassing rates of about  $2 \times 10^{-8}$  and  $5 \times 10^{-8}$  torr litres  $\text{cm}^{-2} \text{sec}^{-1}$  respectively, but at the same time during the second cycle the rate of evolution was already about  $10^{-10}$  torr litres  $\text{cm}^{-2} \text{sec}^{-1}$ .

Observations made using a large sample of  $3750 \text{ cm}^2$  separately evacuated and attached to the mass spectrometer ionization chamber showed the outgassing at the end of the baking cycle to be over 90% hydrogen with traces of  $\text{CO}$ ,  $\text{H}_2\text{O}$ , and  $\text{CO}_2$ . Comparison of the composition and quantity of gas being evolved at the time of maximum evolution with that at the end of the cycle can be made by reference to the mass spectra shown in Figs. 14 and 15 which were made during the initial bake-out of sample 2. Here again hydrogen is shown to be the final component (See also Calder and Lewin<sup>(4)</sup>).

A graph, Fig. 16, presenting log outgassing versus  $\frac{1}{T}$  made during the cooling of the large specimen after sixteen hours bake at  $450^{\circ}\text{C}$  produced a straight line with a slope consistent with that obtained from an activated diffusion process for hydrogen with an

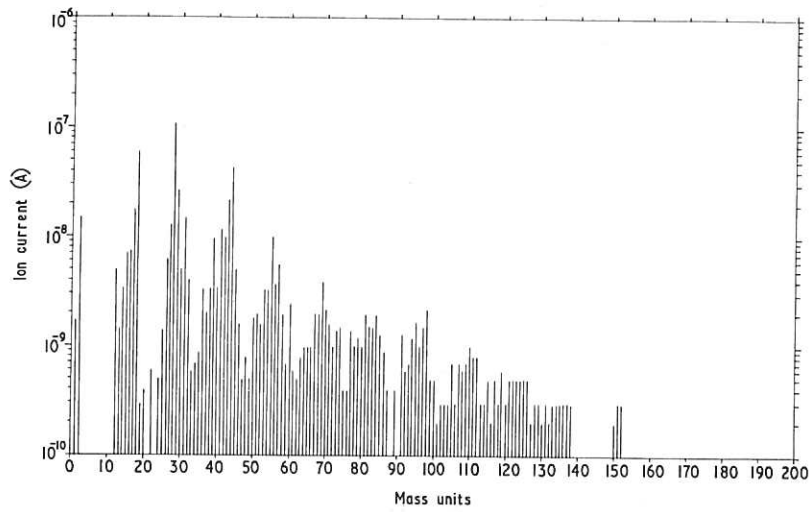


Fig. 14 (CLM-R 93)  
 Sample 2. 1st bake-out. Temp. 285°C 1.75 hrs after commencing outgassing. Expt. 25. Date 29-30/11/66. Surface finish 31  $\mu$  in

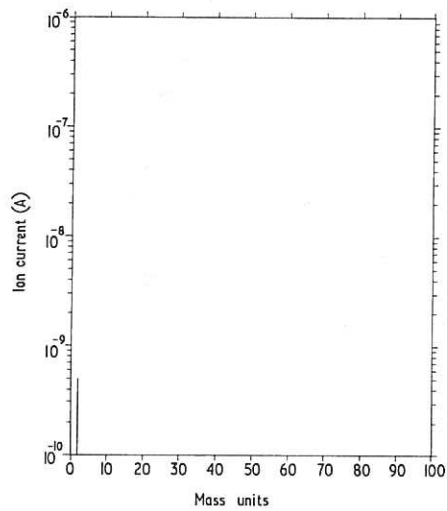


Fig. 15 (CLM-R 93)  
 Sample 2. 1st bake-out. Temp. 450°C 20 hrs after commencing bake. Expt. 25. Date 29-30/11/66. Surface finish 31  $\mu$  in

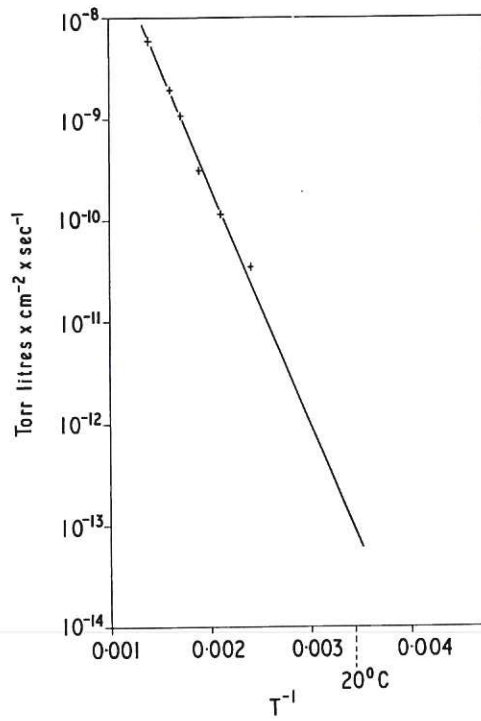


Fig. 16 (CLM-R 93)  
 Dependence of the outgassing rate of stainless steel on temp.  
 Expt. 10 (S2). Sample 2. Date 5/1/1968

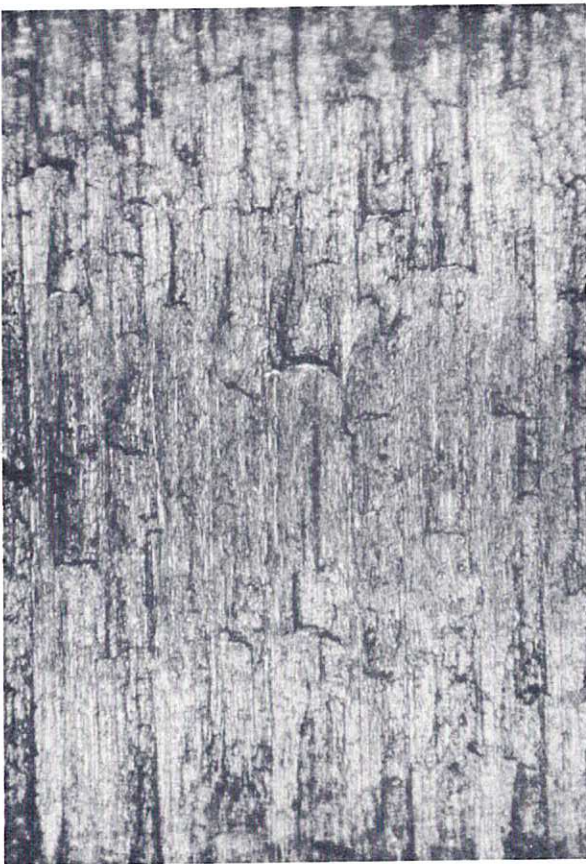


Fig. 17 (CLM-R 93)  
 Surface of dry machined sample showing tearing. Magnification x 100

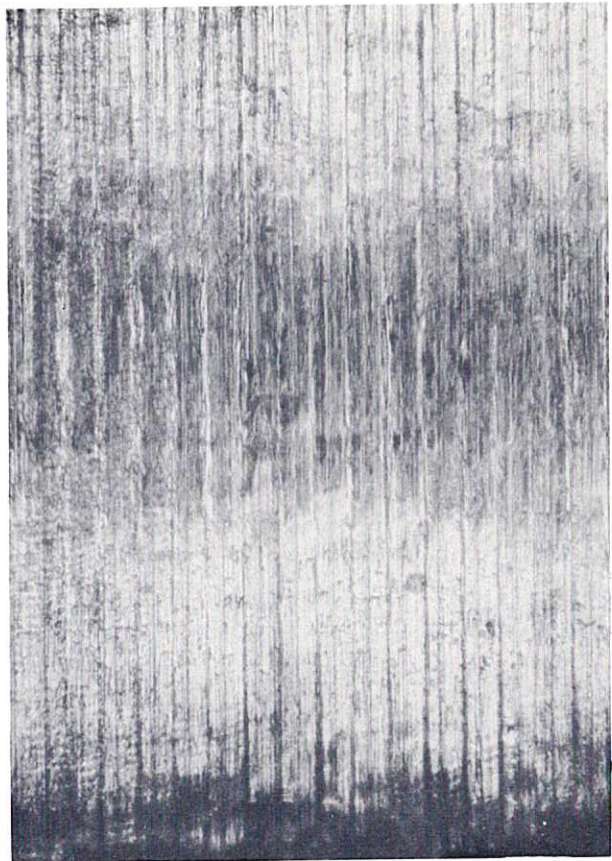


Fig. 18 (CLM-R 93)  
 Surface of sample machined using cutting oil. Magnification x 100



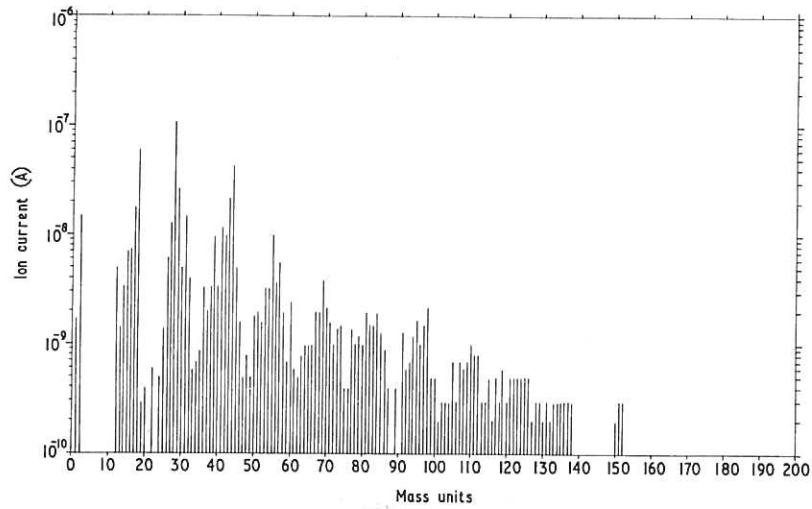


Fig. 14 (CLM-R 93)  
 Sample 2. 1st bake-out. Temp. 285°C 1.75 hrs after commencing outgassing. Expt.25. Date 29-30/11/66. Surface finish 31  $\mu$  in

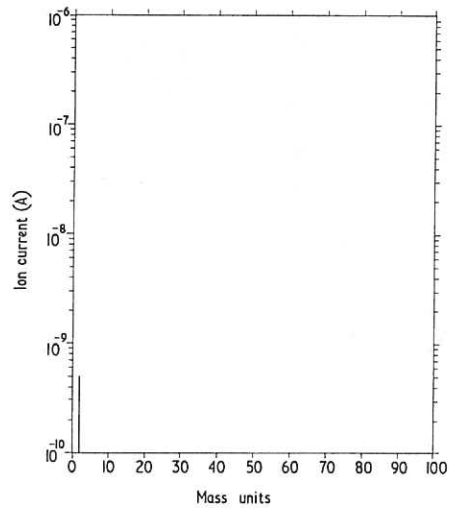


Fig. 15 (CLM-R 93)  
 Sample 2. 1st bake-out. Temp.450°C 20 hrs after commencing bake. Expt. 25. Date 29-30/11/66. Surface finish 31  $\mu$  in

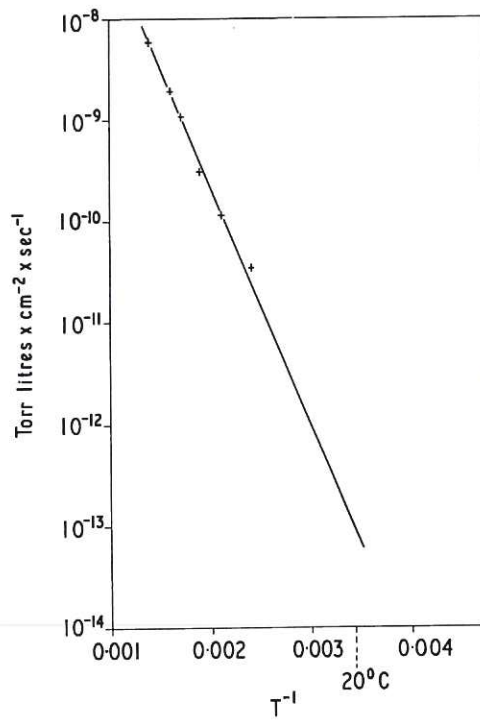


Fig. 16 (CLM-R 93)  
 Dependence of the outgassing rate of stainless steel on temp.  
 Expt. 10 (S2). Sample 2. Date 5/1/1968

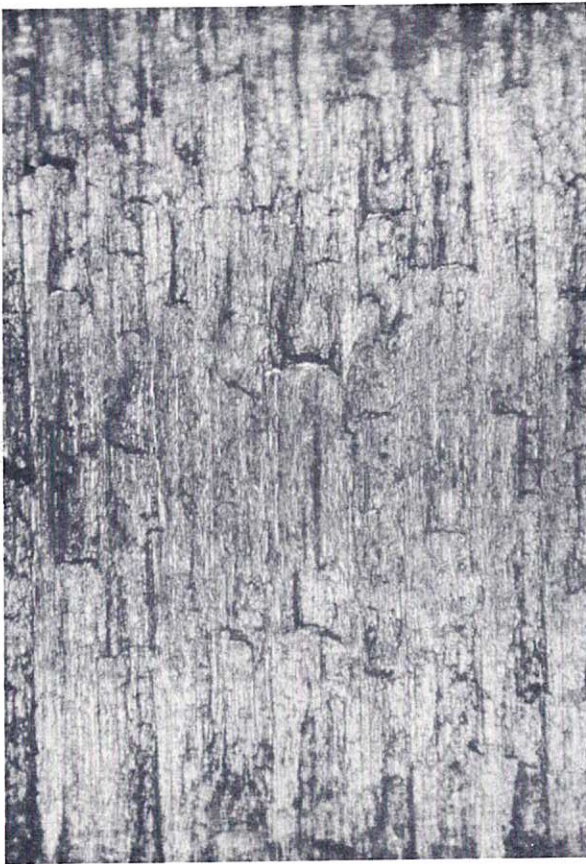


Fig. 17 (CLM-R 93)  
 Surface of dry machined sample showing tearing. Magnification x 100

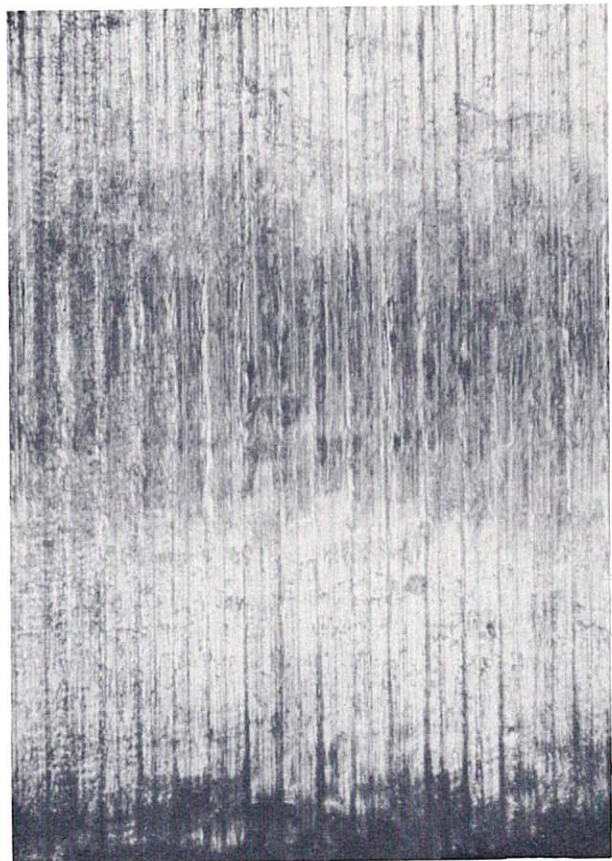


Fig. 18 (CLM-R 93)  
 Surface of sample machined using cutting oil. Magnification x 100

activation energy of 10 kcal/mole. This figure is in good agreement with the findings of Jones et al.<sup>(5)</sup> for EN58E stainless steel. Extrapolation of this curve shows an evolution rate of about  $10^{-13}$  torr litres  $\text{cm}^{-2}$   $\text{sec}^{-1}$  at room temperature.

#### Source of Gas

If it is accepted that the gas released up to a temperature of about 320°C is mainly surface adsorbed then reference to Figs.3,5,7,9,11 and 13 and Table 2 gives good evidence that a very large percentage of the gas evolved by the clean specimens following exposure to atmosphere was a result of surface adsorption, while information obtained from bake-out of the large sample and from the later stages during the baking of standard samples supports the opinion that the remaining gas is primarily hydrogen diffusing from the interior of the metal.

#### Specific Surface

Acceptance of the above concept would infer that the surface available for gas adsorption is much greater than that measured geometrically even allowing for the machine finish and errors of pressure measurement. This is readily seen by reference to Table 1 which shows up to 26 monolayers of gas evolution by clean samples following exposure to atmosphere. In the case of the coarse and dry machined samples, which showed 26 and 22 monolayers respectively, this may be partly explained by tearing of the surface during machining as seen in Fig.17, which shows the surface of sample 1 (dry machined) magnified 100 times. Sample 2 (machined using lubricant) however, which appeared free from tearing (Fig.18) showed only half the gas evolution. The high figure of 7 monolayers obtained from the electropolished surface is not at present explained. It is nevertheless of interest to compare it with the result quoted by Schram<sup>(6)</sup> of 1.2 - 1.3 for the specific surface of electro polished stainless steel NS22S and 2.8 for sheet of the same type.

#### Possibility of contamination by cleaning solvent

The present arrangement, in which gas from the sample entered the ionization chamber without collision with a cold surface presented good conditions under which to study the possibility that traces of trichloroethylene remained as a surface contaminant after bake.

Traces were occasionally observed during the early stages of the initial bake-out cycle (Fig.19), but these were rapidly obscured by the general hydrocarbon background and had fallen below the minimum pressure detectable by the mass spectrometer when this had been removed. Similar results were obtained using acetone and isopropyl alcohol.

#### Efficiency of re-adsorbed gas removal at 200°, 300° and 450°C

The efficiency of removal of the re-adsorbed gas on the surface of a sample by baking in vacuo at 200° and 300°C in relation to a similar bake at 450°C was studied. A sample outgassed for 16 hours at 450°C was exposed, on cooling, to atmosphere for twenty-four hours after which it was baked at 200°C in the mass spectrometer for sixteen hours. At the end of the period the temperature was raised to 450°C. The gas released during the period at 200° was then measured as a percentage of the total gas evolved. A similar set of observations was made at 300°C in place of 200°. The results showed, Fig.20, that only 40% of the total gas evolved was released at 200°C, but that the bake at 300°C, Fig.21, released 70%.

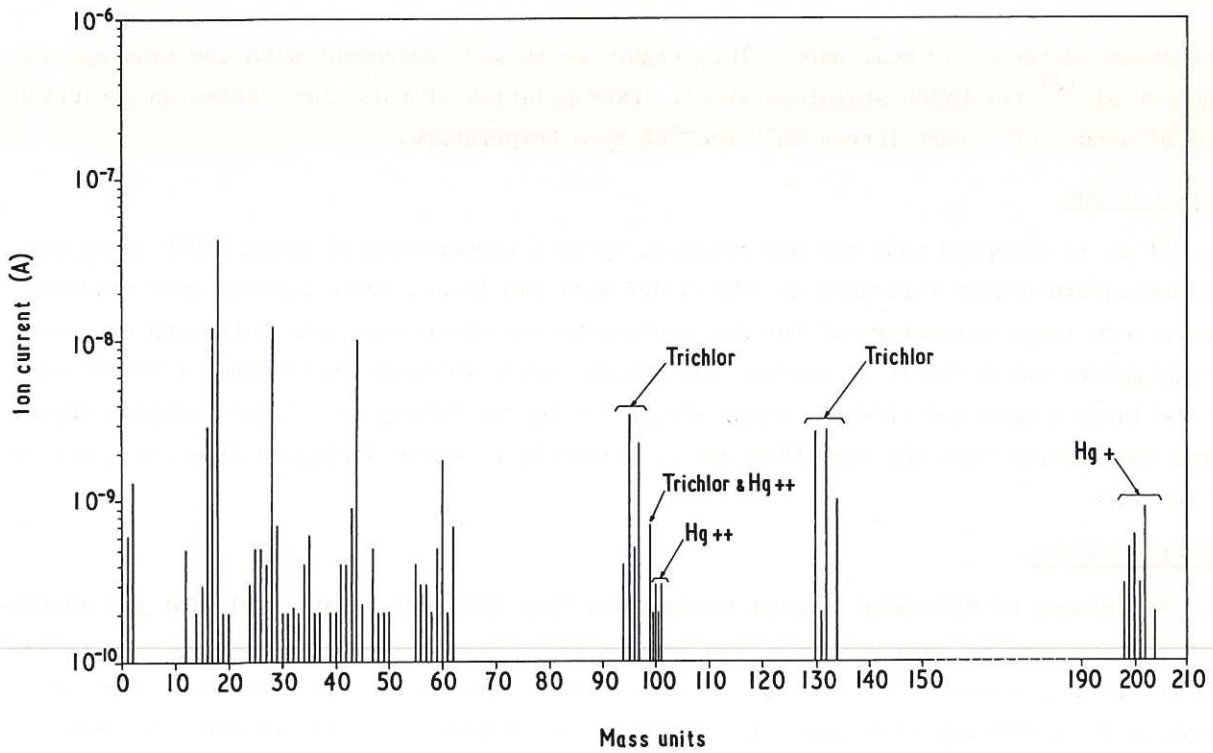


Fig. 19 (CLM-R 93)  
 Mass spectrum showing trichlorethylene peaks seen during early stages of first bake-out. Temp. 85°C. Expt. 1. Date 27-28/4/66

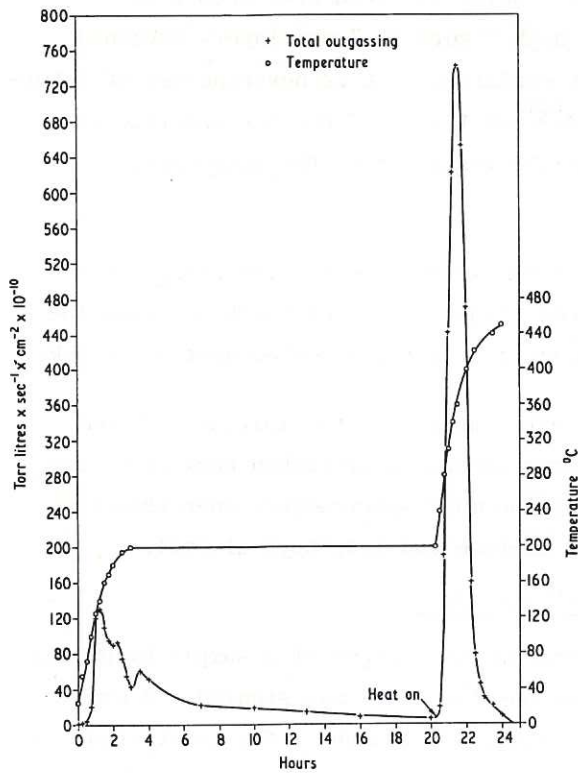


Fig. 20 (CLM-R 93)  
 Bake-out of sample (previously baked at 450°C in vacuo) following exposure to atmos. Matl. 18/8/1. Expt. 42. Sample 25 Date 20-22/3/67. Max. temp. 200°C. (1st stage) 450°C (2nd stage). Surface finish 18 μ in

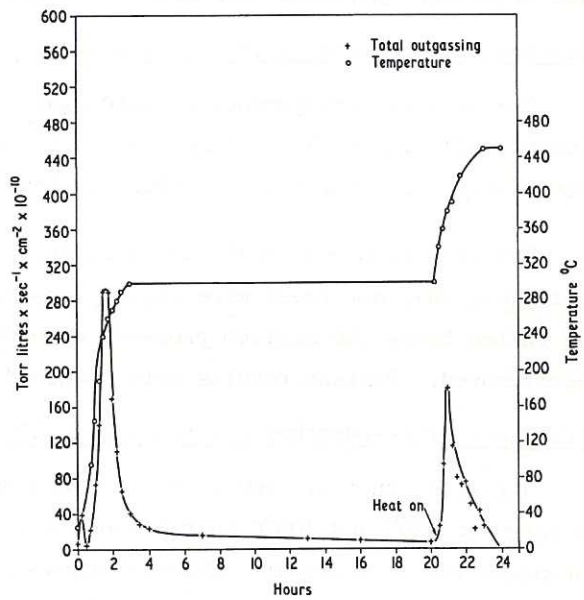


Fig. 21 (CLM-R 93)  
 Bake-out of sample (previously baked at 450°C in vacuo) following exposure to atmos. Matl. 18/8/1. Expt. 43. Sample 25 Date 19/4/67. Max temp. 300°C (1st stage) 450°C (2nd stage) Surface finish 18 μ in.

## 6. CONCLUSIONS

An initial bake-out of new 18/8/1 stainless steel ultra high vacuum components at 450°C in vacuo successfully removes all gases resulting from the previous history with the exception of traces of hydrogen. There is good evidence that this remaining gas is diffusing from the bulk of the metal.

Gas adsorbed by the surface of the stainless steel on re-exposure to atmosphere can be removed by baking at 450°C for a period of 2-3 hours. For many purposes in the  $10^{-9}$  torr region sufficient gas can be removed by a 12 hour bakeout from 200-300°C. In such cases it is worthwhile arranging for new components to be baked at 450°C in a separate vacuum oven before assembly into the apparatus, thus avoiding the need to design the plant to withstand such high temperature.

The methods of cleaning tested, while capable of efficiently removing gross contamination were not comparable with vacuum bake-out in the reduction of outgassing. Surface finish plays an important part in the outgassing rate once the initial soils have been removed by the initial bake and high standards of machining are advised, preferably an electropolished surface if this can be obtained without danger of permanent trapping of solution in crevices.

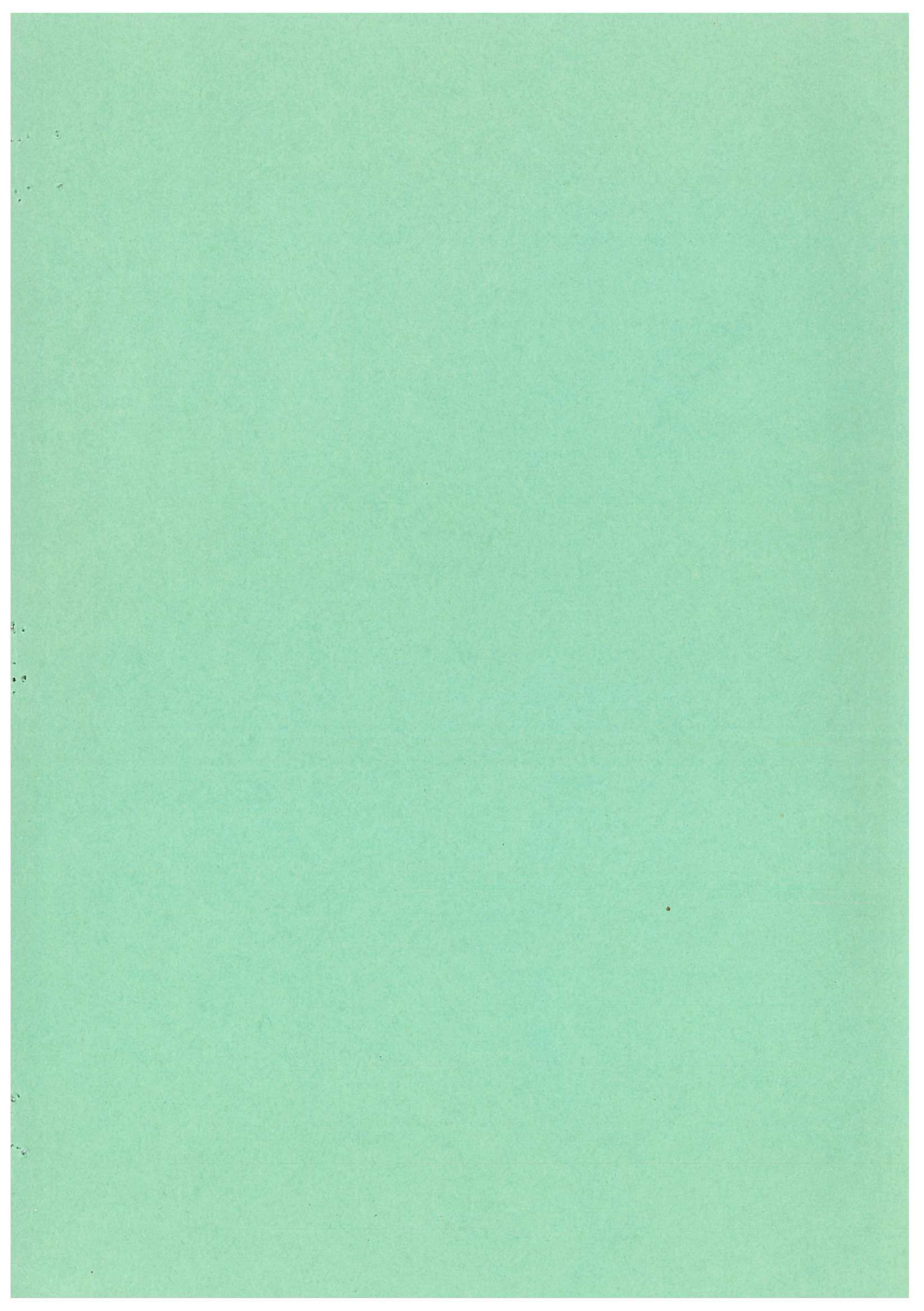
## 7. ACKNOWLEDGEMENTS

The authors are grateful to Dr G.M. McCracken and Mr H.H.H. Watson for advice.

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