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TEGNIESE / TECHNICAL MEMORANDUM NO. 36 of 1968.

INTERIM REPORT ON THE CHARACTERISTICS OF THE VOLATILE MATTER OF COALS.

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cosmi Coal Mining Research Controlling Council

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INTRODUCTION:

The object of this investigation was to determine whether a correlation could be found between the known explosive properties of coal dusts and the nature, quantity and temperature dependence of volatile matter formation.

A preliminary study of the nature of the volatile matter formed when coal is pyrolysed was described in F.R.I. Technical Memorandum No. 39 of 1967. The platinum wire spiral used to heat the coal in the preliminary experiments proved too fragile and results were difficult to reproduce. The pyrolysis temperature could not be determined or controlled accurately. Further pyrolysis experiments were therefore carried out in a small electrically heated tubular oven under closely controlled conditions of temperature and duration. The results using the improved heating technique show excellent reproducibility.

Although it was not possible to reproduce the fast rate of heating which prevails during an explosion, the results obtained here should have some bearing upon the explosion process, especially in regard to the nature and amount of volatile matter present in coal.

EXPERIMENTAL.

The pyrolysis oven was constructed in the laboratory from readily available materials. A suitable length of ¼ inch

stainless...../

stainless steel tubing served as the oven. One end of the tubing was threaded to fit the injection port of the gas chromatograph and the other end fitted with a tee-piece through which the coal sample could be introduced and the carrier gas (Helium) flowed. A 3 inch length of the tubing was enclosed in an electrically heated sleeve - the temperature of which was determined by means of a calibrated thermocouple. A nickel sample boat was attached permanently to a wire pushrod which passed through a gas-tight seal in the tee-piece. A weighed sample of coal spread evenly on the bottom of the sample boat to ensure rapid heat transfer, could be pushed into the hot part of the oven and withdrawn at will.

The volatile pyrolysis products are flushed into the gas chromatograph where they are separated according to boiling point. Components with boiling points of up to 350°C can be detected. Tarry matter with a higher boiling point is not eluted from the chromatograph. It is not possible to detect hydrogen, carbon monoxide or carbon dioxide as the flame ionization detector of the gas chromatograph only registers the presence of organic compounds.

RESULTS.

Pyrolysis was carried out at 250°C, 350°C, 450°C 500°C and 550°C on freshly ground 10 mg. portions of minus 100 mesh coal. Chromatograms of the volatile matter produced when four coals were thus subjected to pyrolysis are reproduced in Figures 1 to 20. The total duration of heating was 10 seconds at the temperature indicated on each chromatogram.

Proximate analyses of the coals, listed in increasing order of explosibility are as follows:

Coal	Ash % (Air-dry)	Moisture % (Air-dry)	Volatile Matter % (Air-dry)	Explosibility
Sigma	29.0	6.6	21.6	Nil
D.N.C.	11.5	1.6	30.2	Weak
Pittsburgh*	4.3	1.5	37.0	Strong
Silkstone**	3.4	1.2	34.5	Strong

* American coal.
** British coal.

Pyrolysis...../

Pyrolysis at the lower temperatures yields the simplest traces. The chromatograms become more complex due to the formation of additional components as the pyrolysis temperature is increased. Appreciable thermal cracking of already formed components is unlikely since they are rapidly swept out of the hot zone of the oven by the inert carrier gas. The ill-defined peaks toward the end of each chromatogram result from the formation of small quantities of tar acids and bases which are not adequately resolved by the chromatographic column.

The prominent peaks on each chromatogram are not necessarily single compounds but can be mixtures of a major component plus one or more minor ones. It is possible to trap any boiling point fraction as it is eluted from the column and subject it to a further analysis. This was not done, however, since all the chromatograms proved similar when considered on a qualitative basis.

Even when differences in volatile matter content are taken into consideration, the coals which are explosive, viz. Silkstone and Pittsburgh, and to a lesser extent D.N.C., yield disproportionately more volatile matter and at a lower temperature than Sigma, which is relatively inert.

A qualitative analysis was made of the atmosphere in the Hartmann bomb after coal dust had been exploded in it, to determine whether any correlation exists between the pyrolysis experiments described above and the actual conditions prevailing during a coal dust explosion. The gas chromatogram reproduced in Figure 21 was the result. The mixture proved unexpectedly complex and apart from the three or four large peaks due to major components a further twenty minor ones were detected. The large peak at the start of the chromatogram is due to methane and possibly acetylene and the prominent one toward the middle was tentatively identified as benzene.

The failure to detect compounds with boiling points appreciably higher than that of benzene is probably due to the sampling method. The procedure was to flush the gaseous mixture in the Hartmann apparatus with helium through a trap cooled in liquid oxygen. Only those compounds with an appreciable vapour pressure at room temperature would be removed from the bomb for further analysis.

It still remains to be proved whether any or all of the large number of organic compounds which were detected are remnants of much larger quantities which partake actively in the explosion, or only minor by-products of no great significance.

Attempts to correlate the volatile matter produced by the low temperature pyrolysis of coals and their explosibility characteristics will be made when information on the high speed high temperature pyrolysis of the coals, which is at present being investigated, becomes available.

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and
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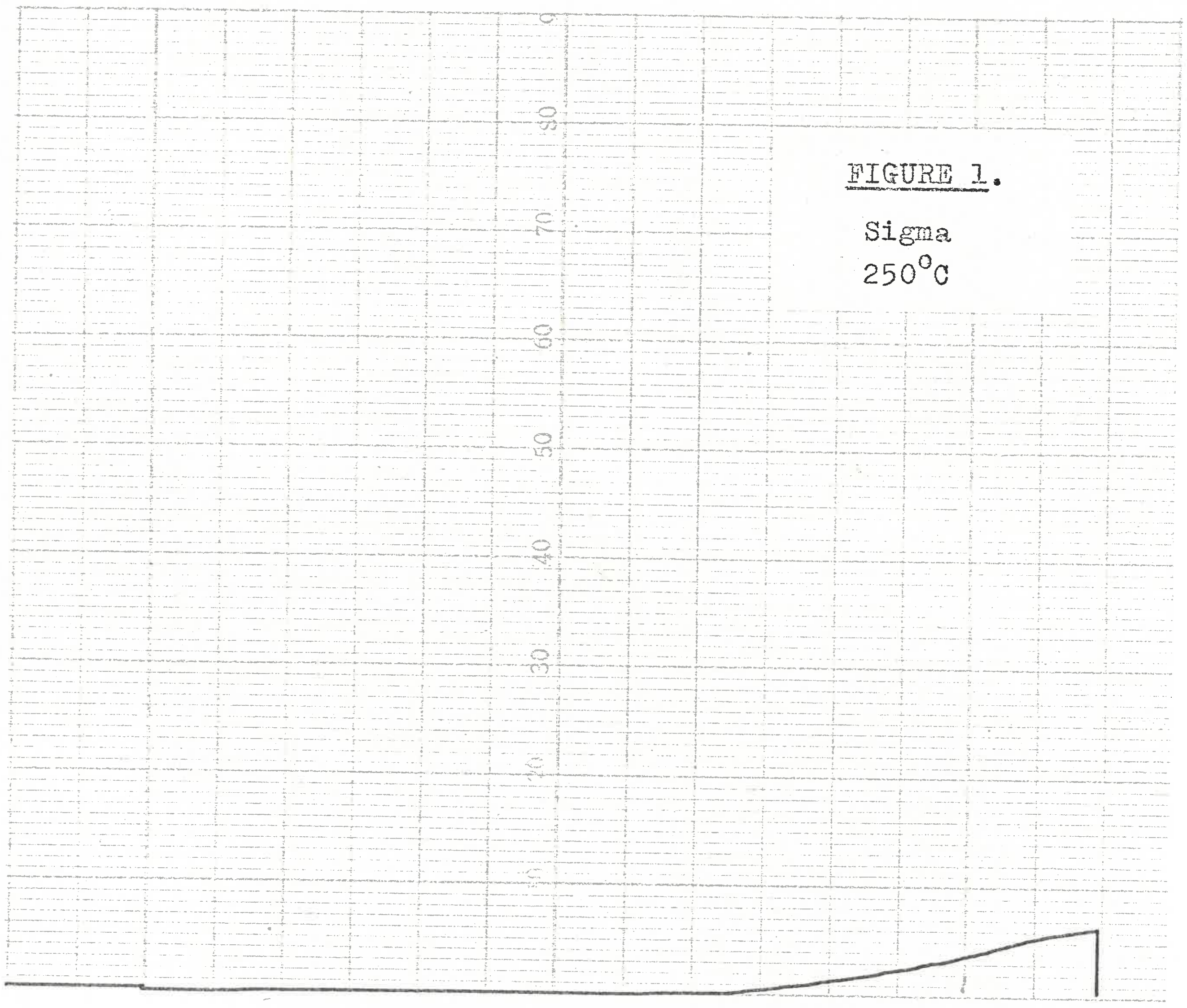
FIGURE 1.
Sigma
250°C

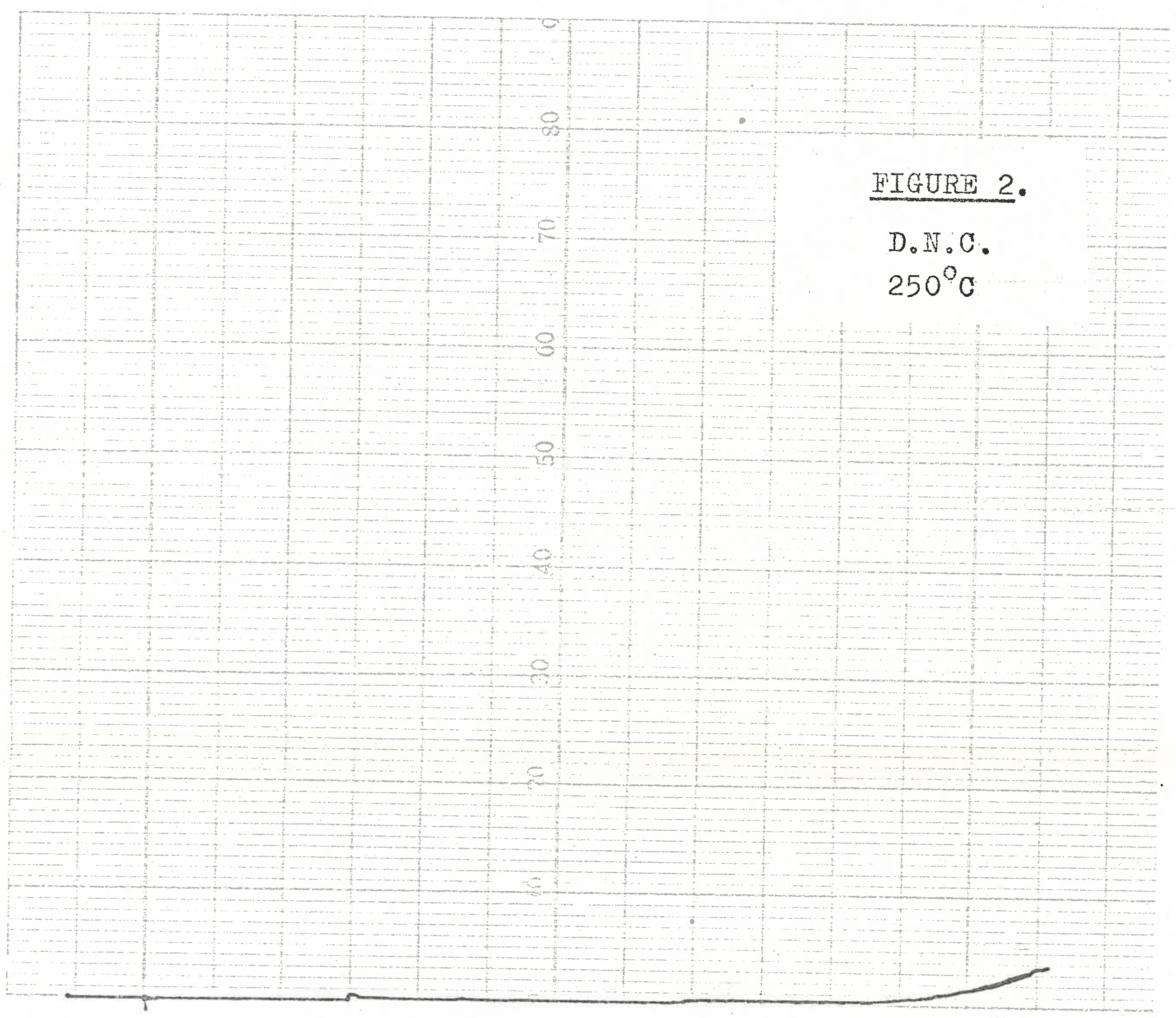
FIGURE 2.
D.N.C.
250°C

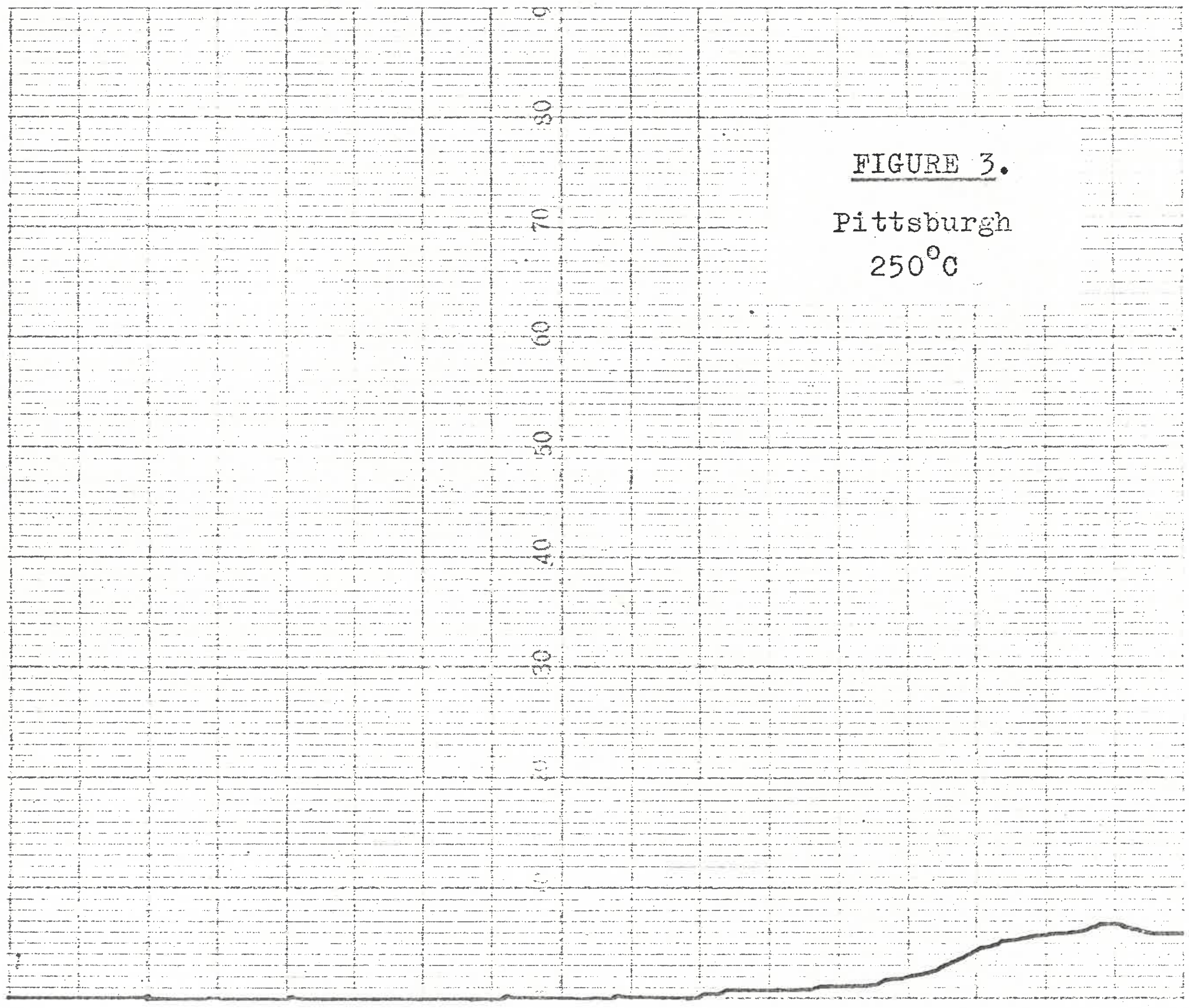

FIGURE 3.
Pittsburgh
250°C

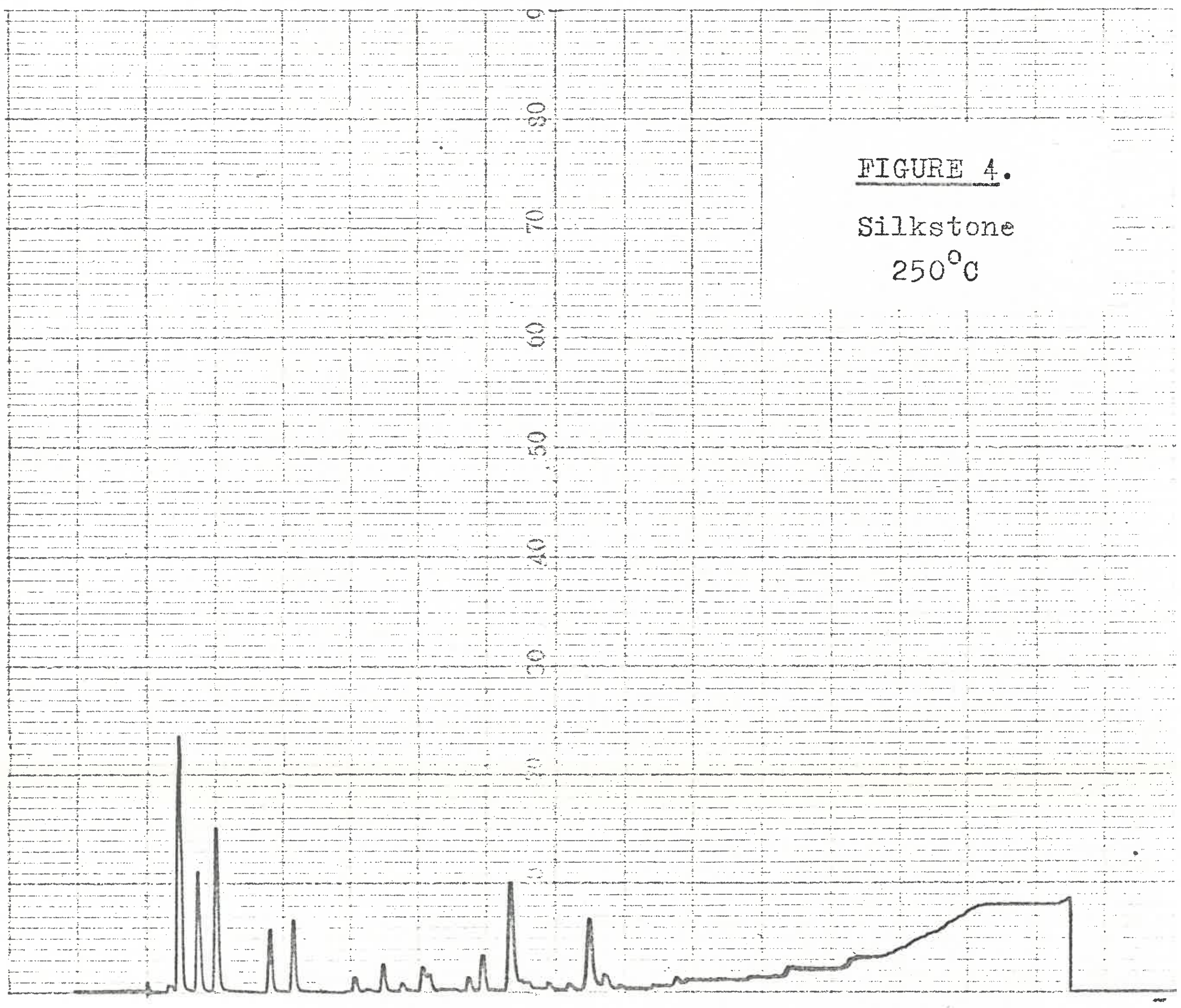

FIGURE 4.
Silkstone
250°C

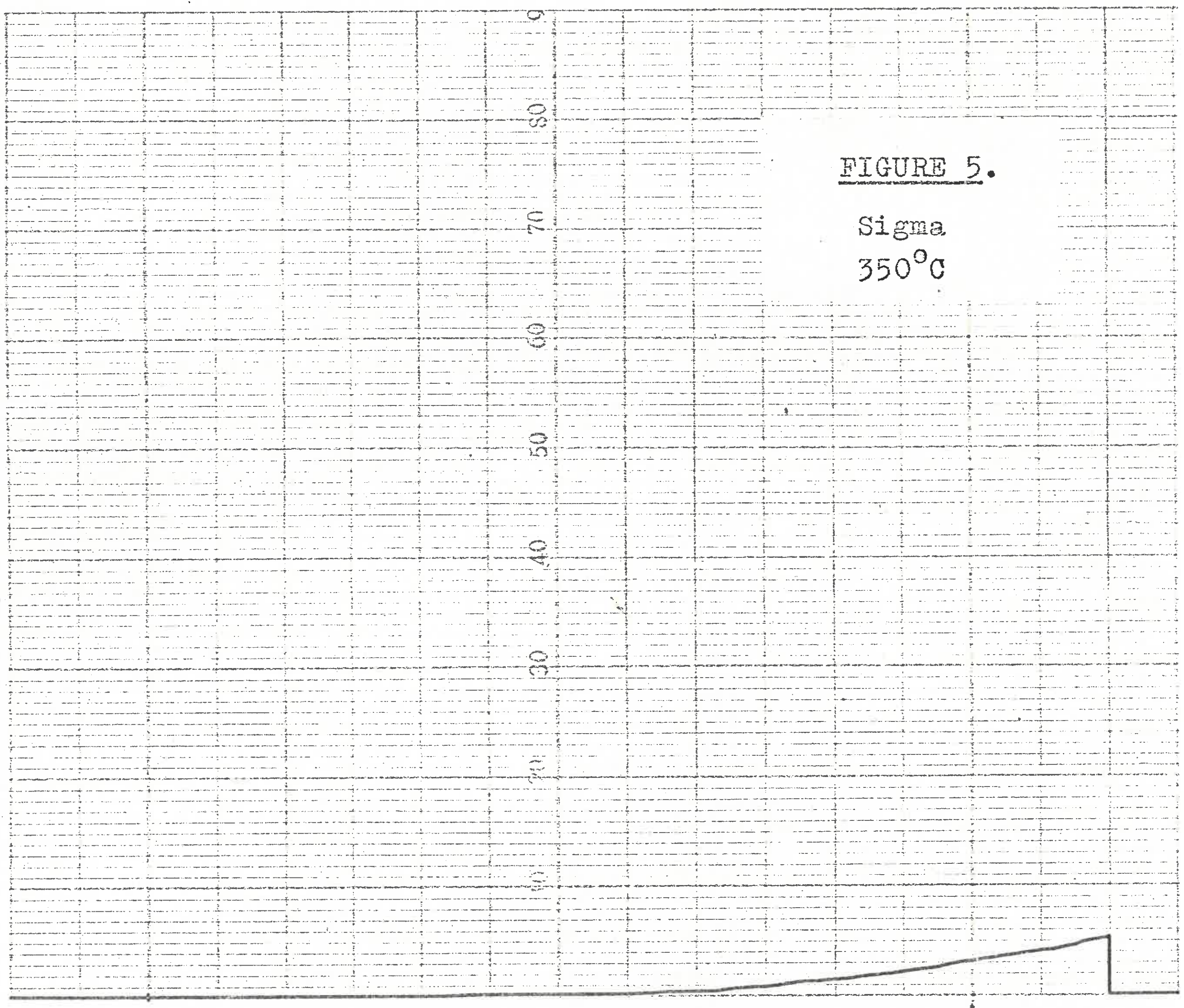

FIGURE 5.

Sigma
$350^{o}C$

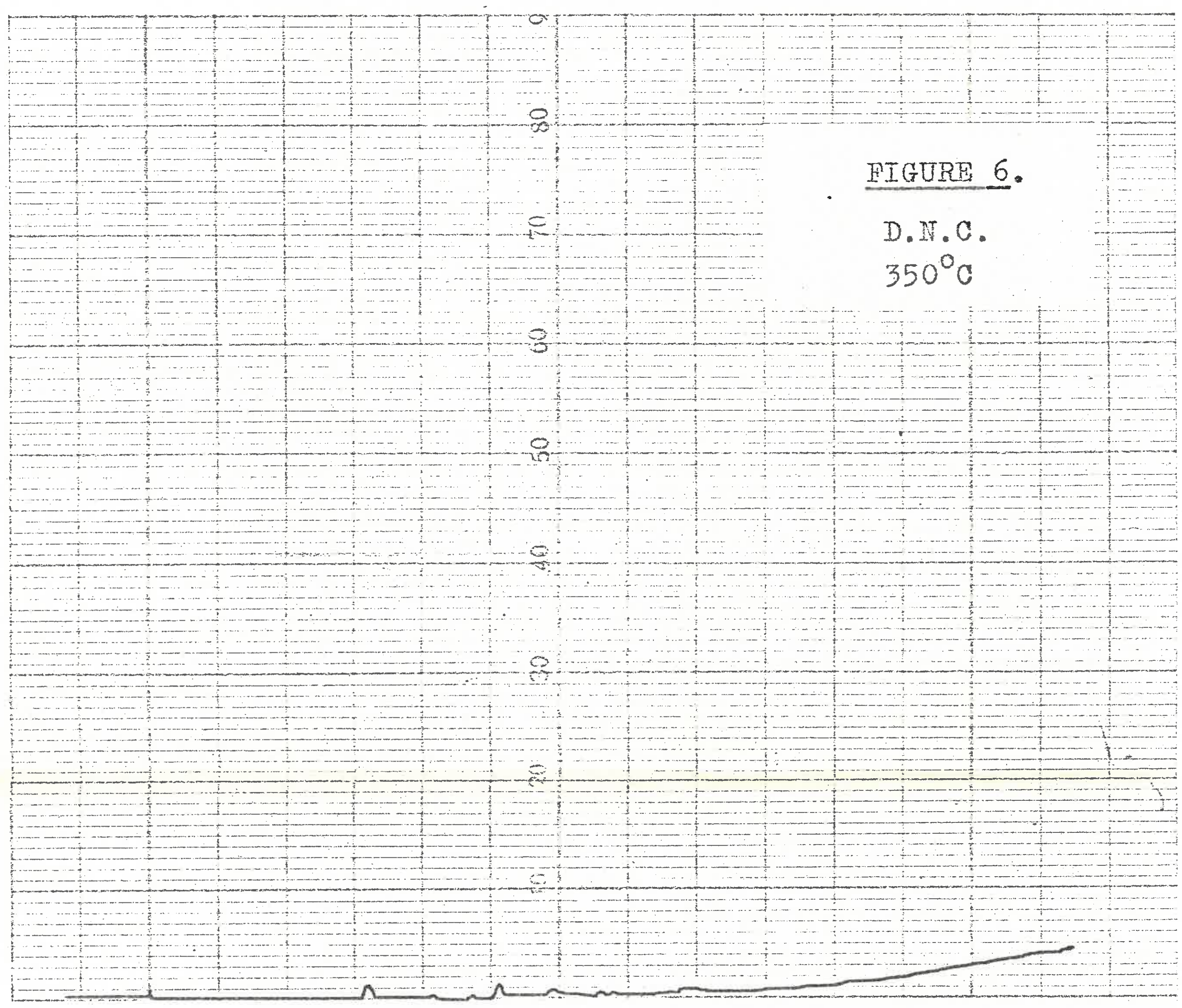

FIGURE 6.

D.N.C.
$350^{o}C$

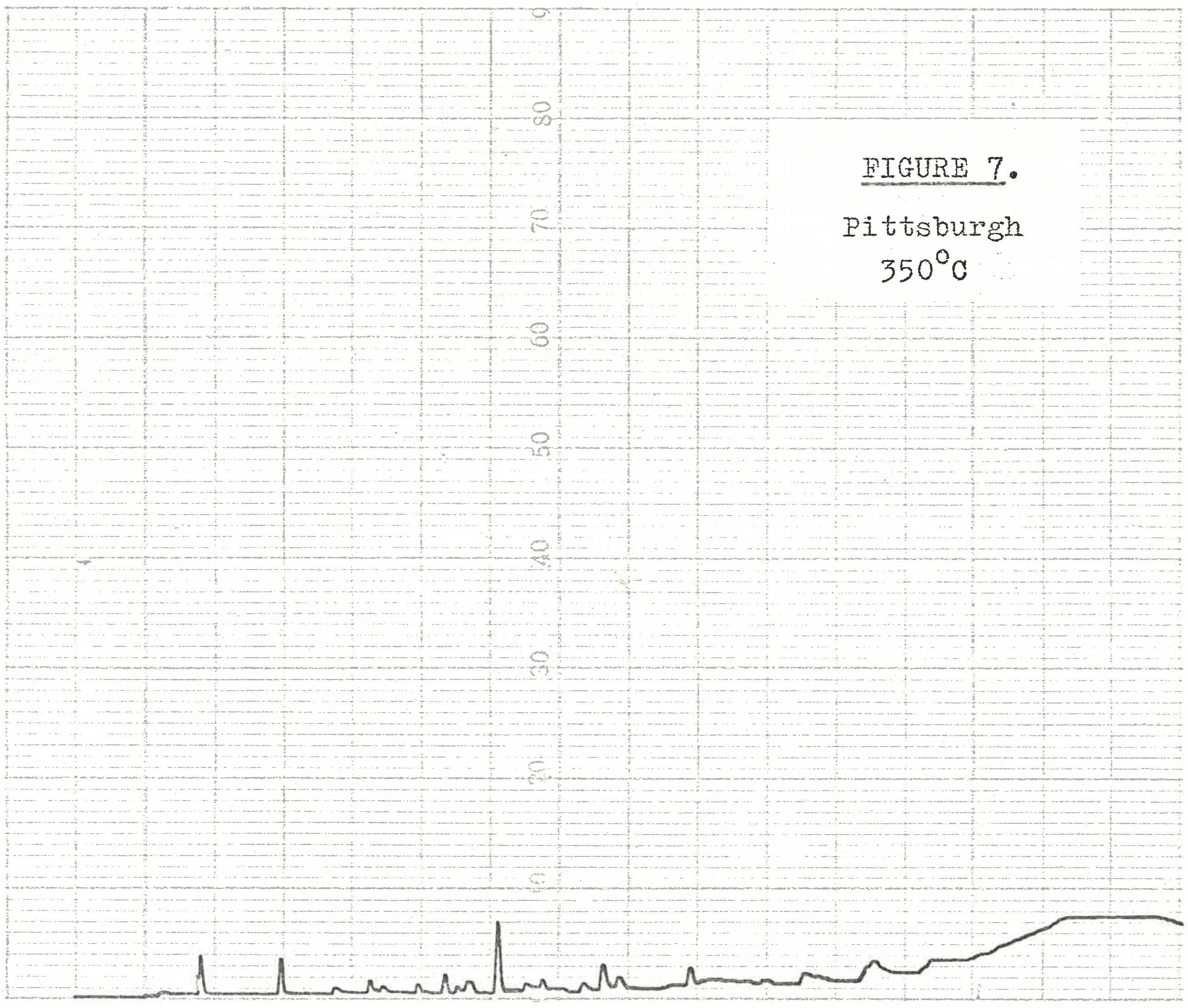

FIGURE 7.

Pittsburgh
350°C

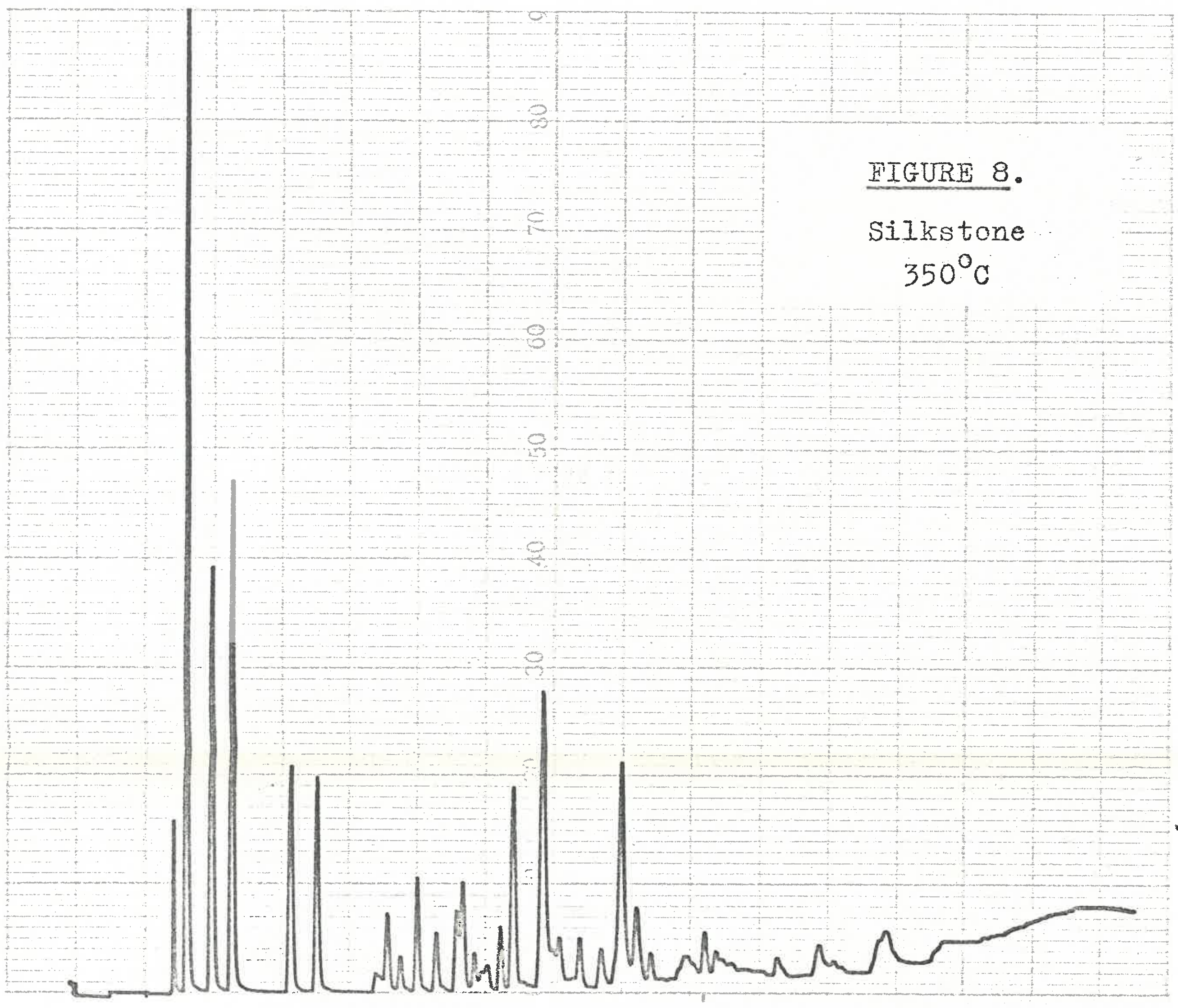

FIGURE 8.

Silkstone
350°C

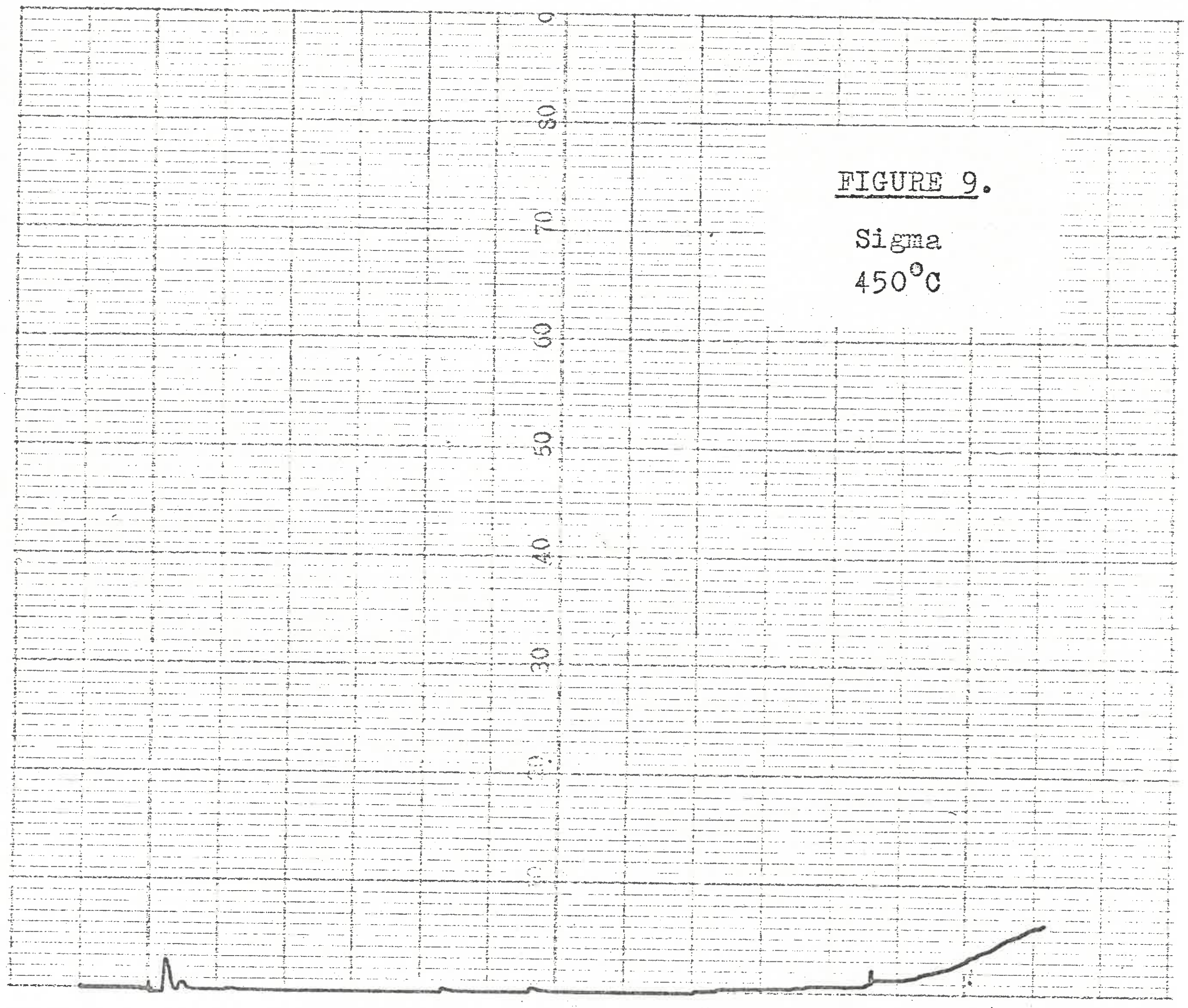

FIGURE 9.

Sigma
450°C

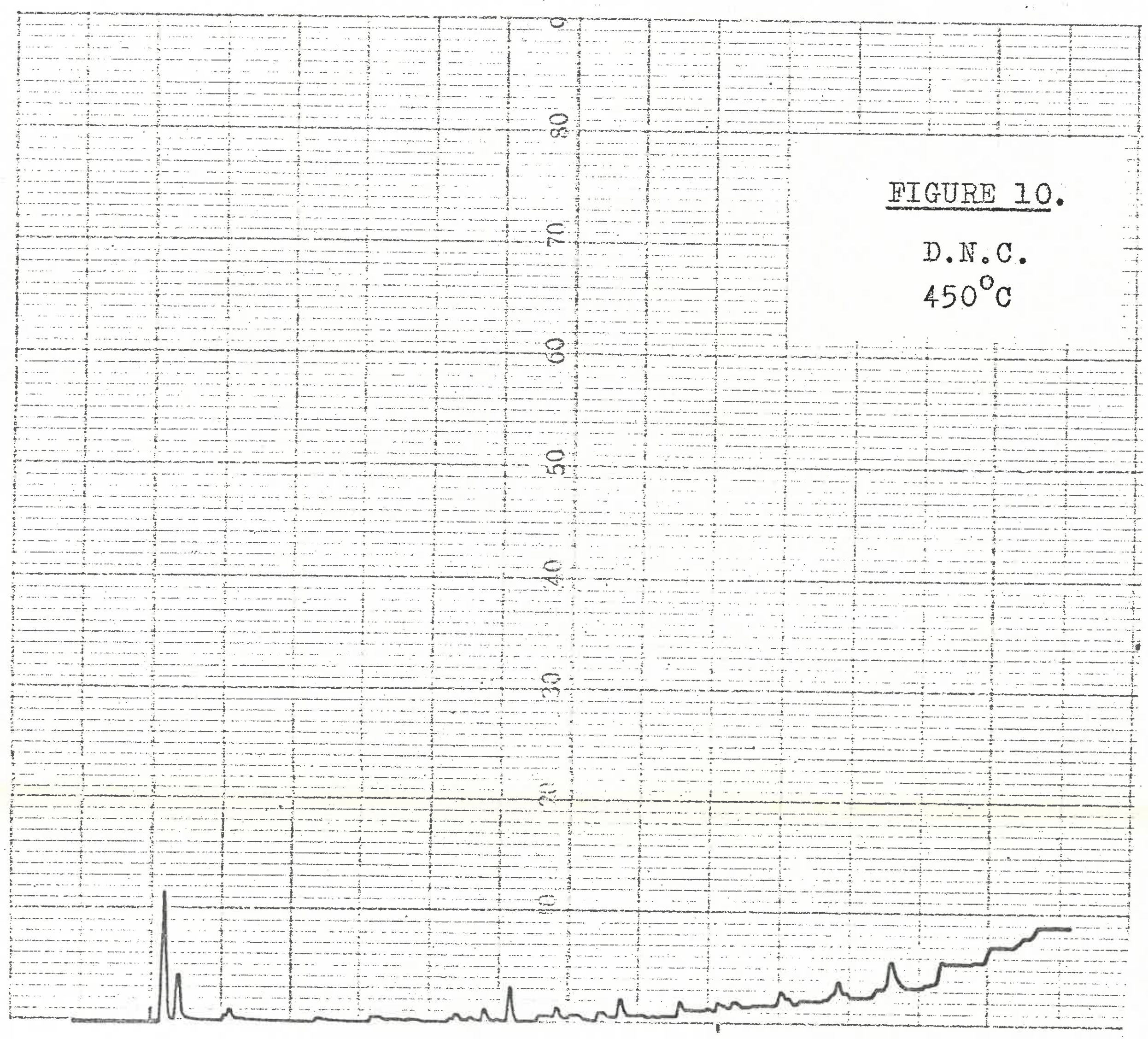

FIGURE 10.

D.N.C.
450°C

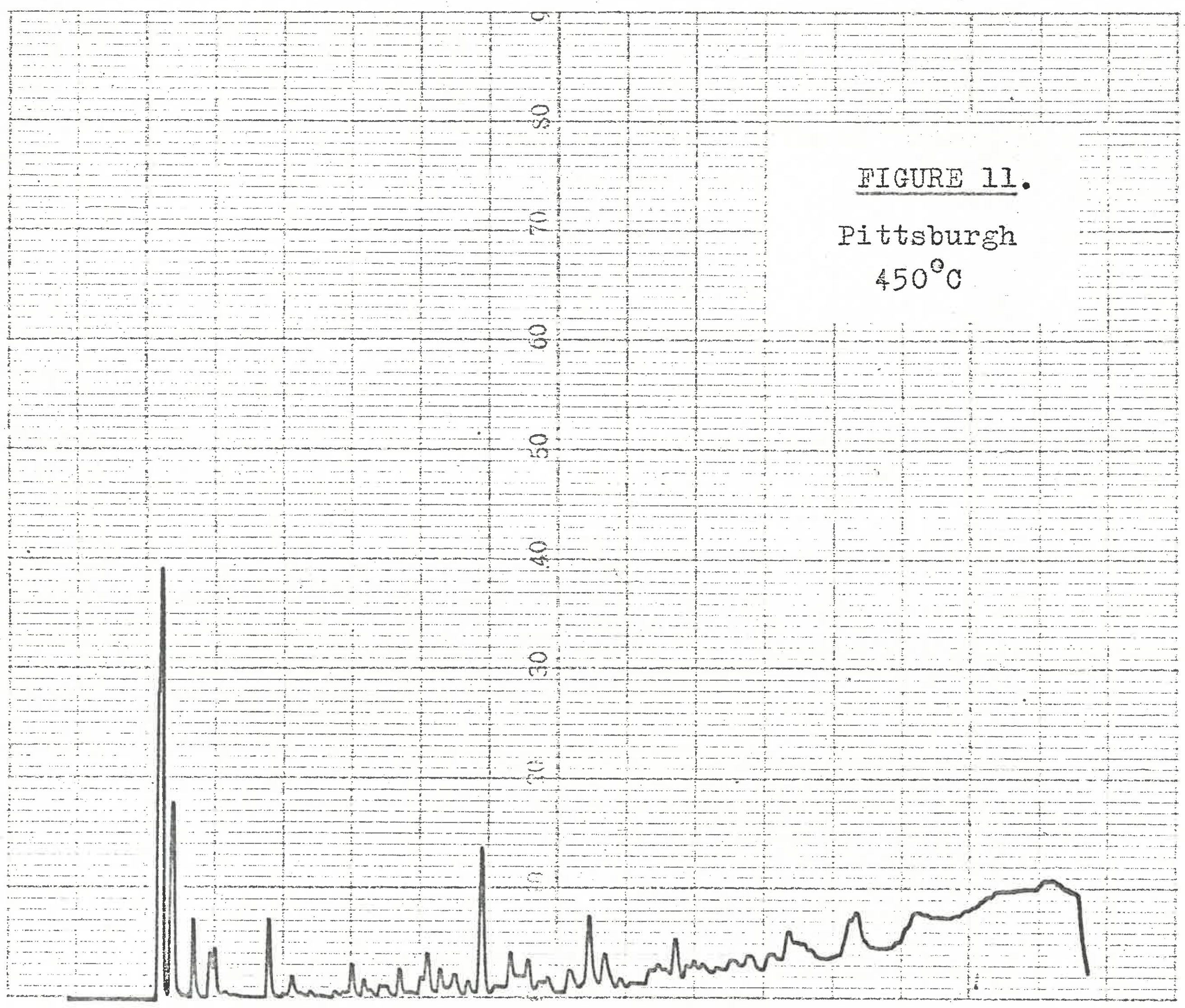

FIGURE 11.

Pittsburgh

450°C

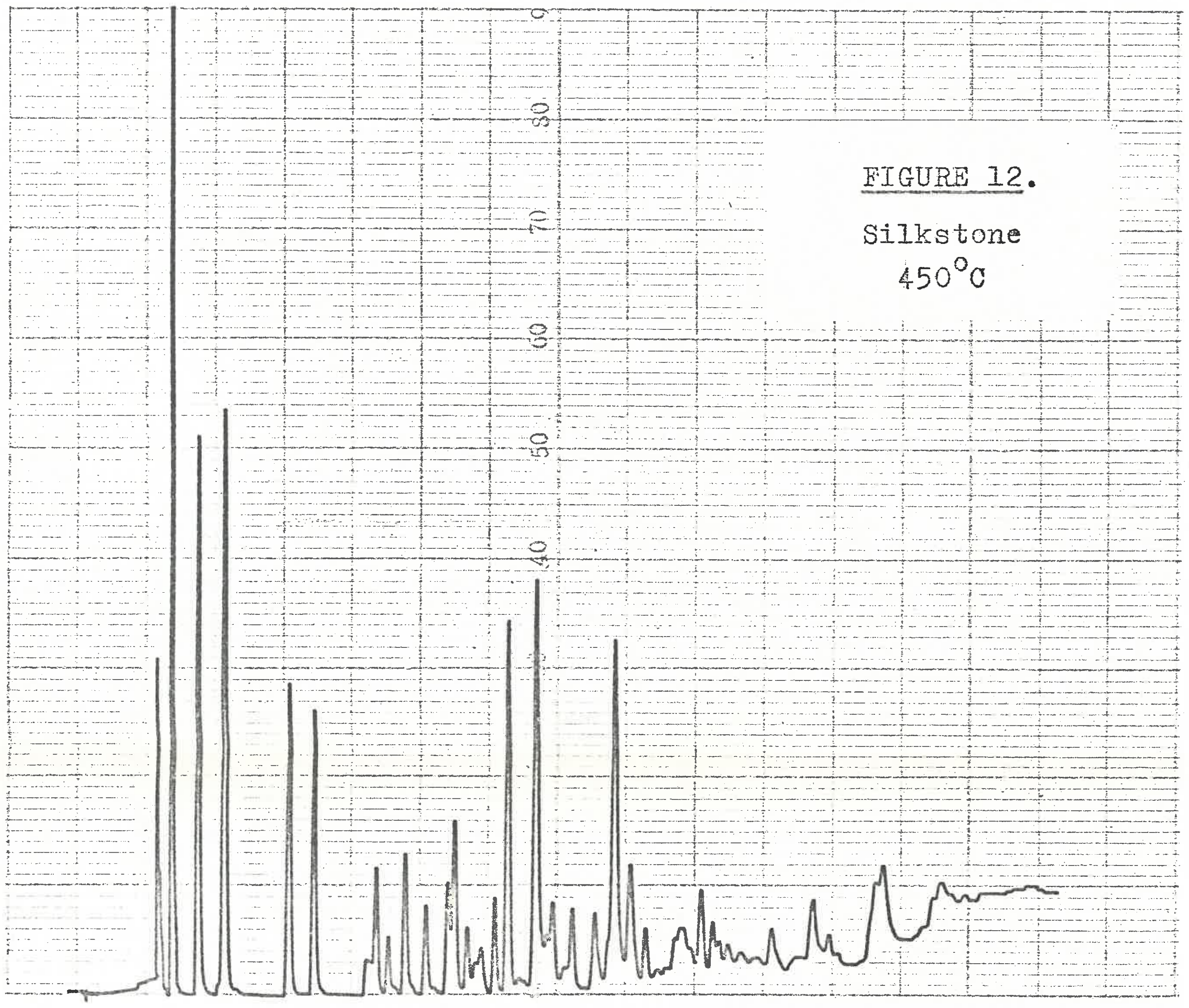

FIGURE 12.

Silkstone

450°C

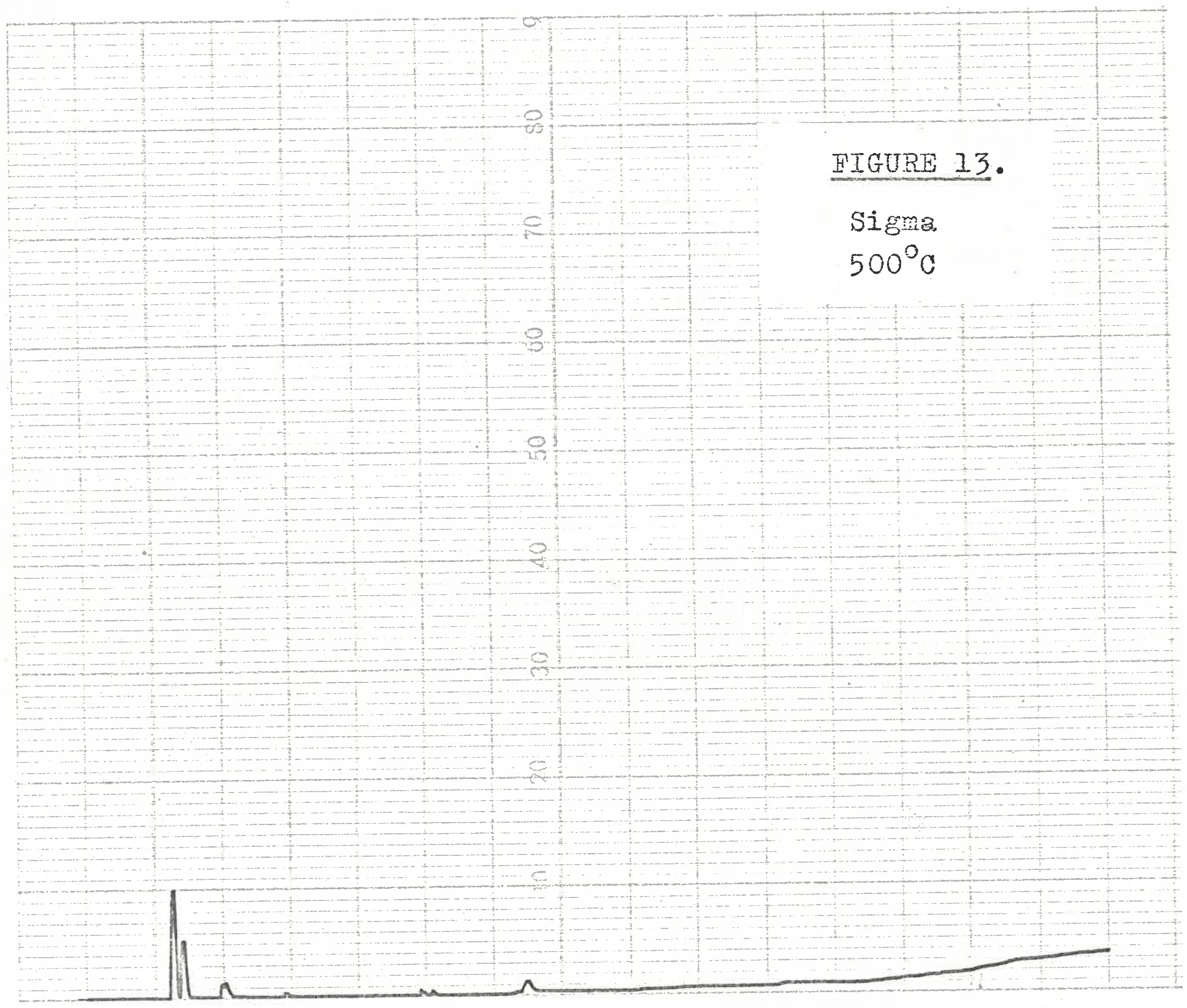

FIGURE 13.

Sigma

500°C

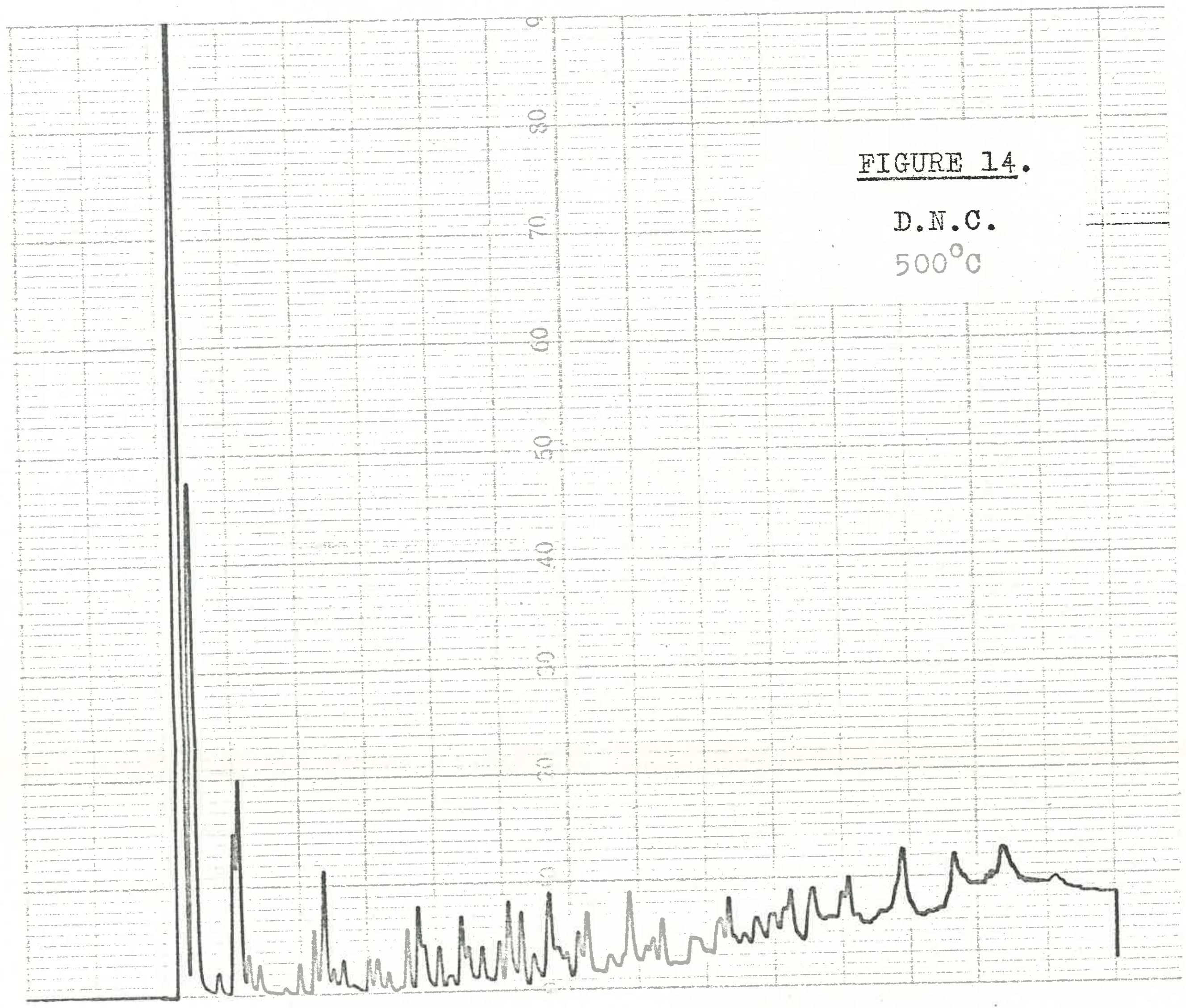

FIGURE 14.

D.N.C.

500°C

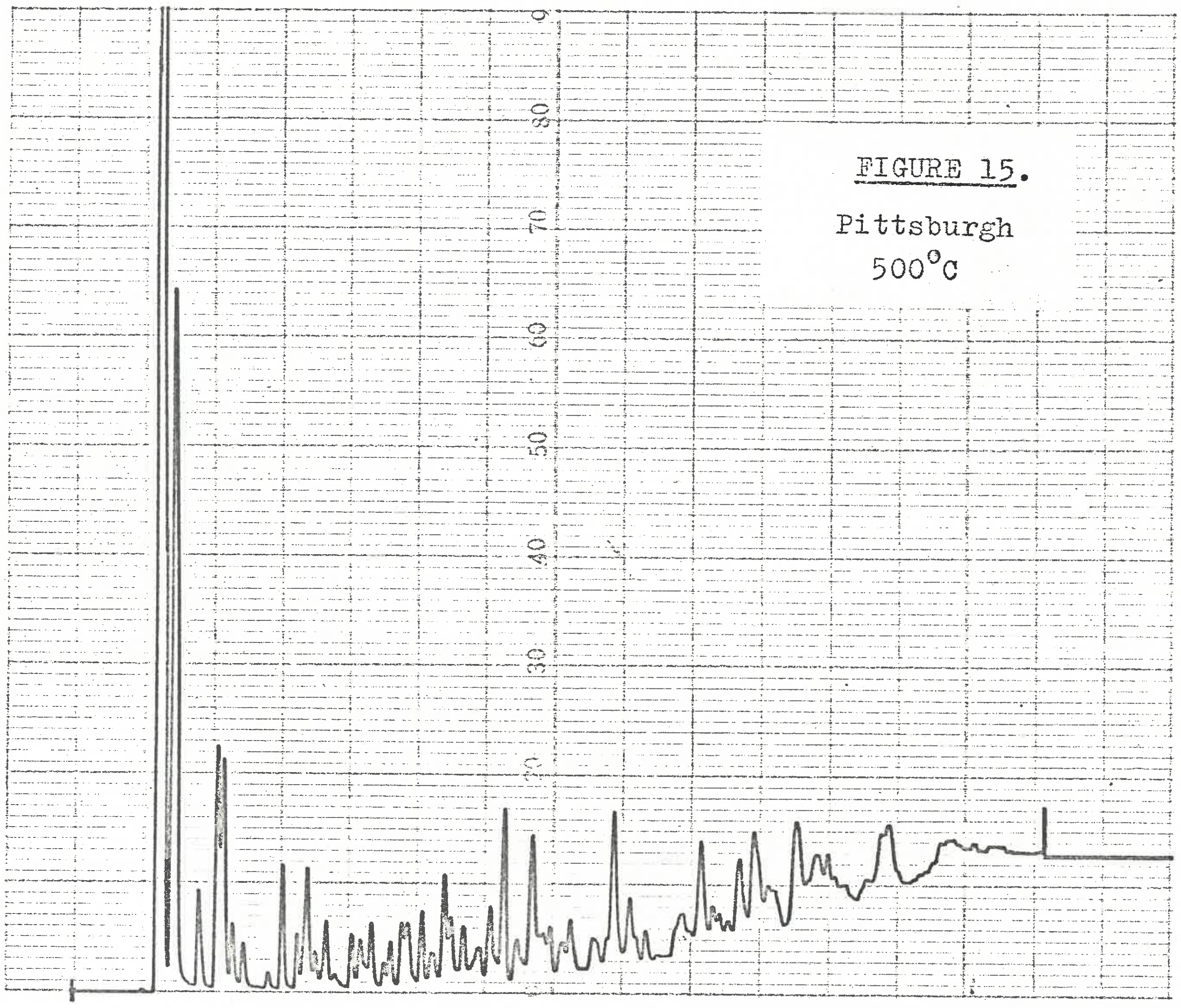

FIGURE 15.

Pittsburgh
500°C

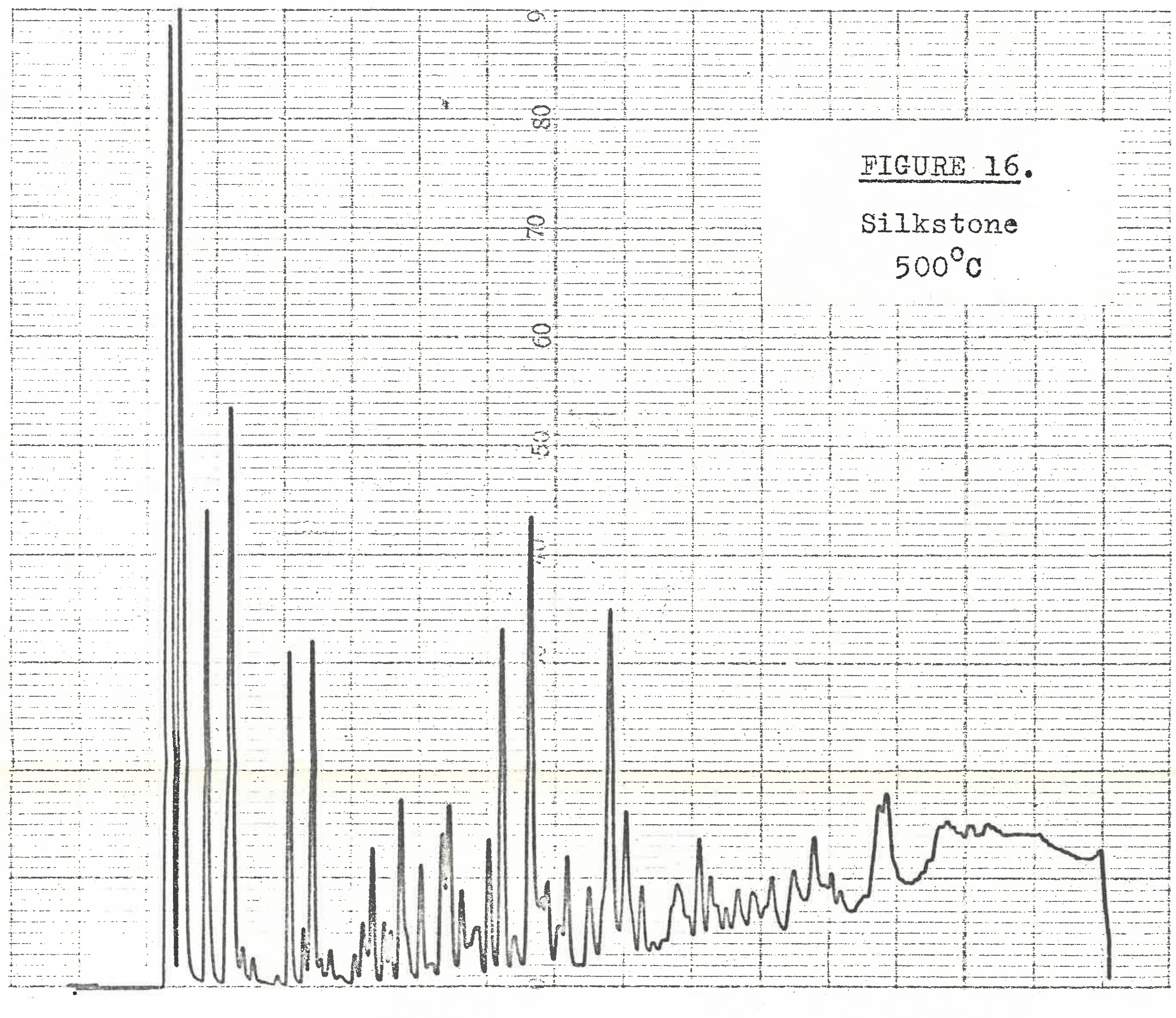

FIGURE 16.

Silkstone
500°C

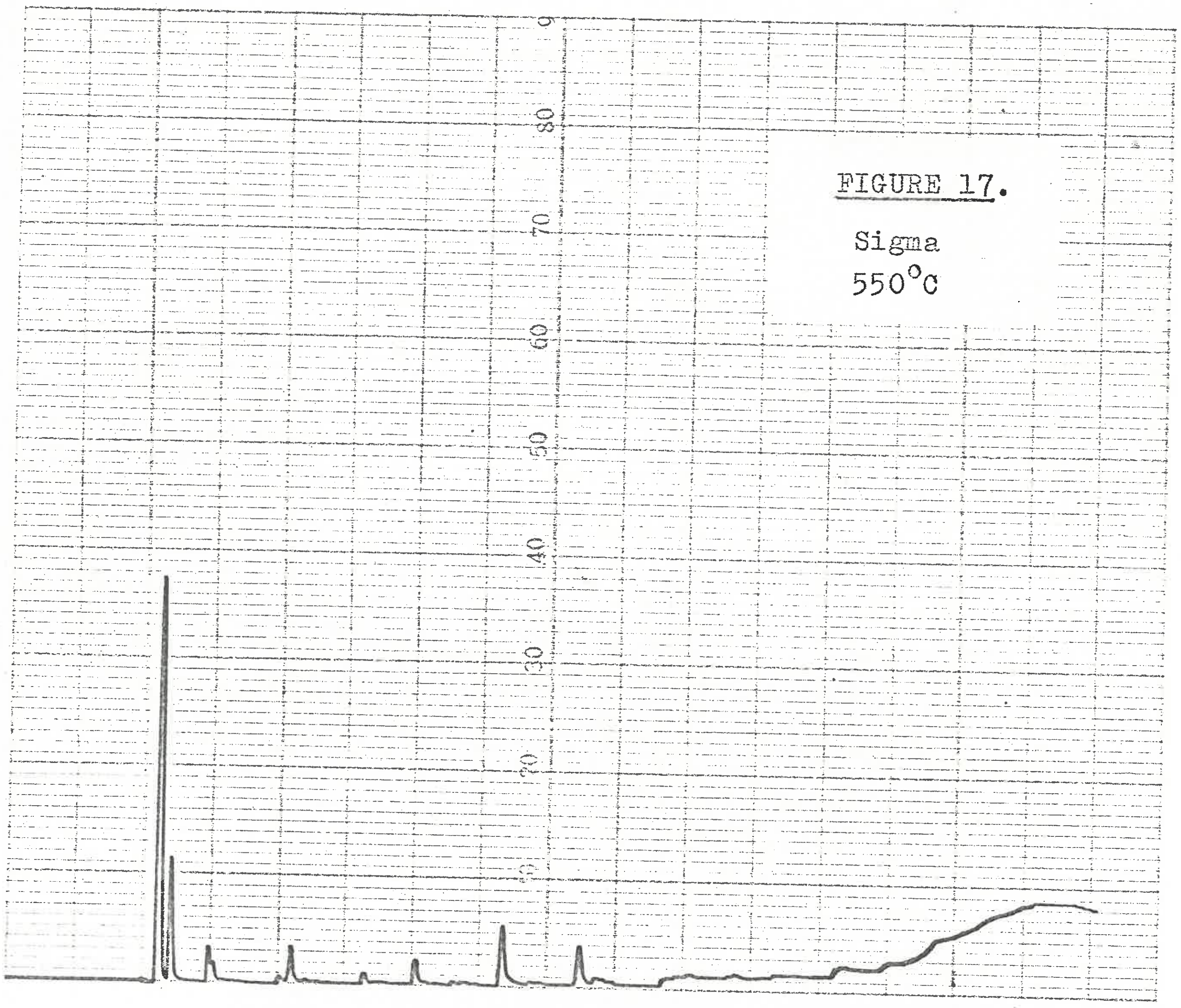

FIGURE 17.

Sigma
550°C

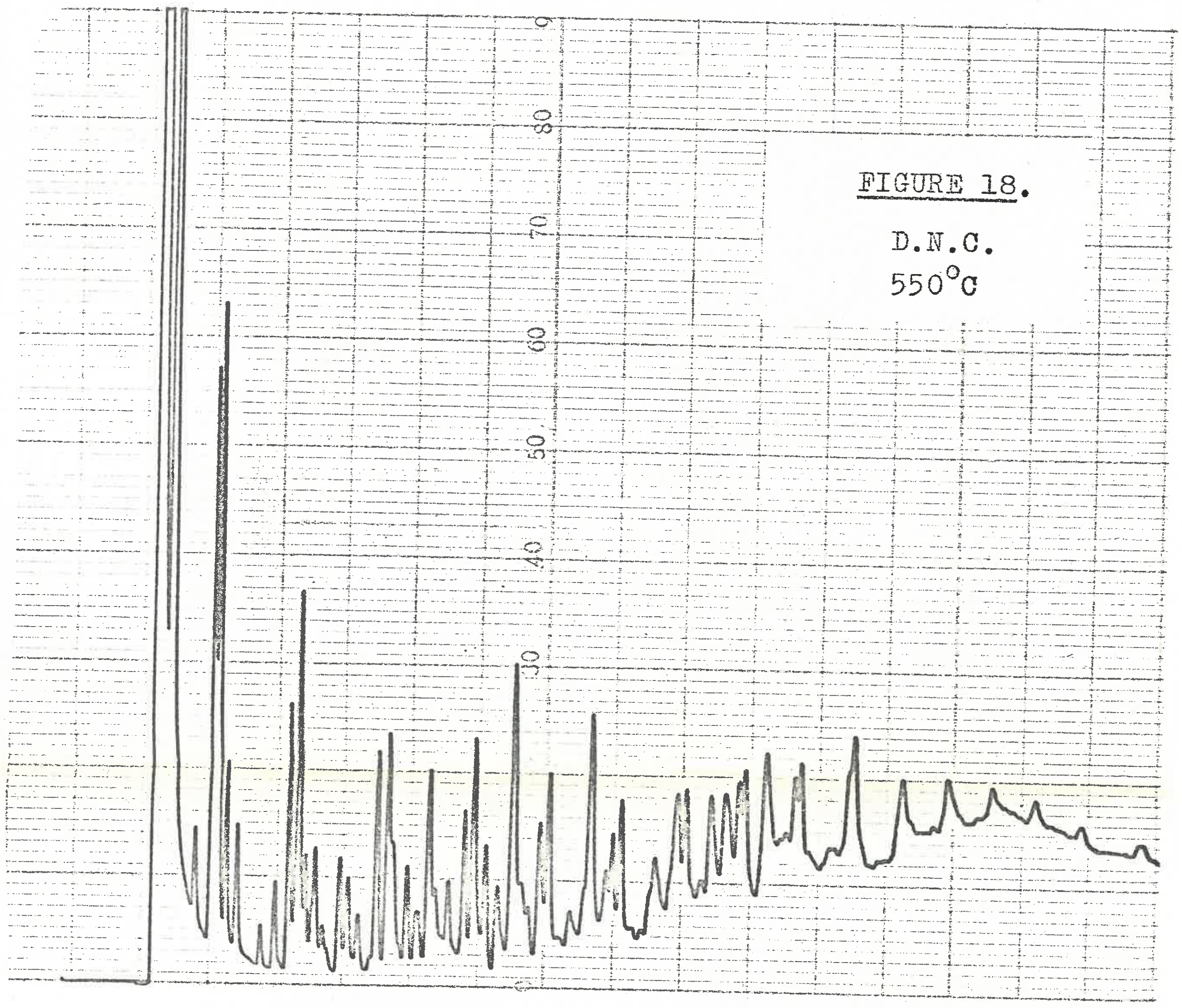

FIGURE 18.

D.N.C.
550°C

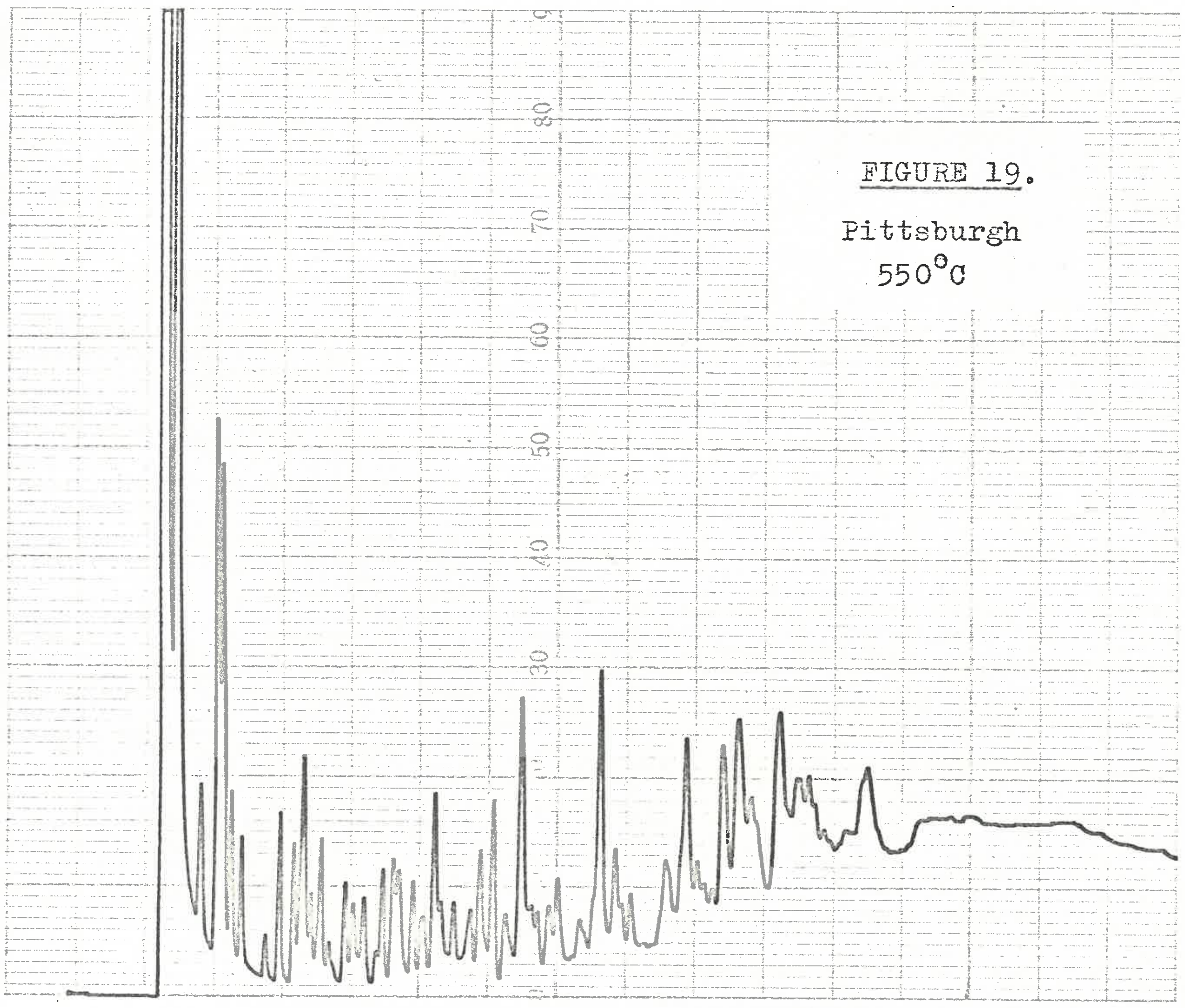
FIGURE 19.
Pittsburgh
550°C

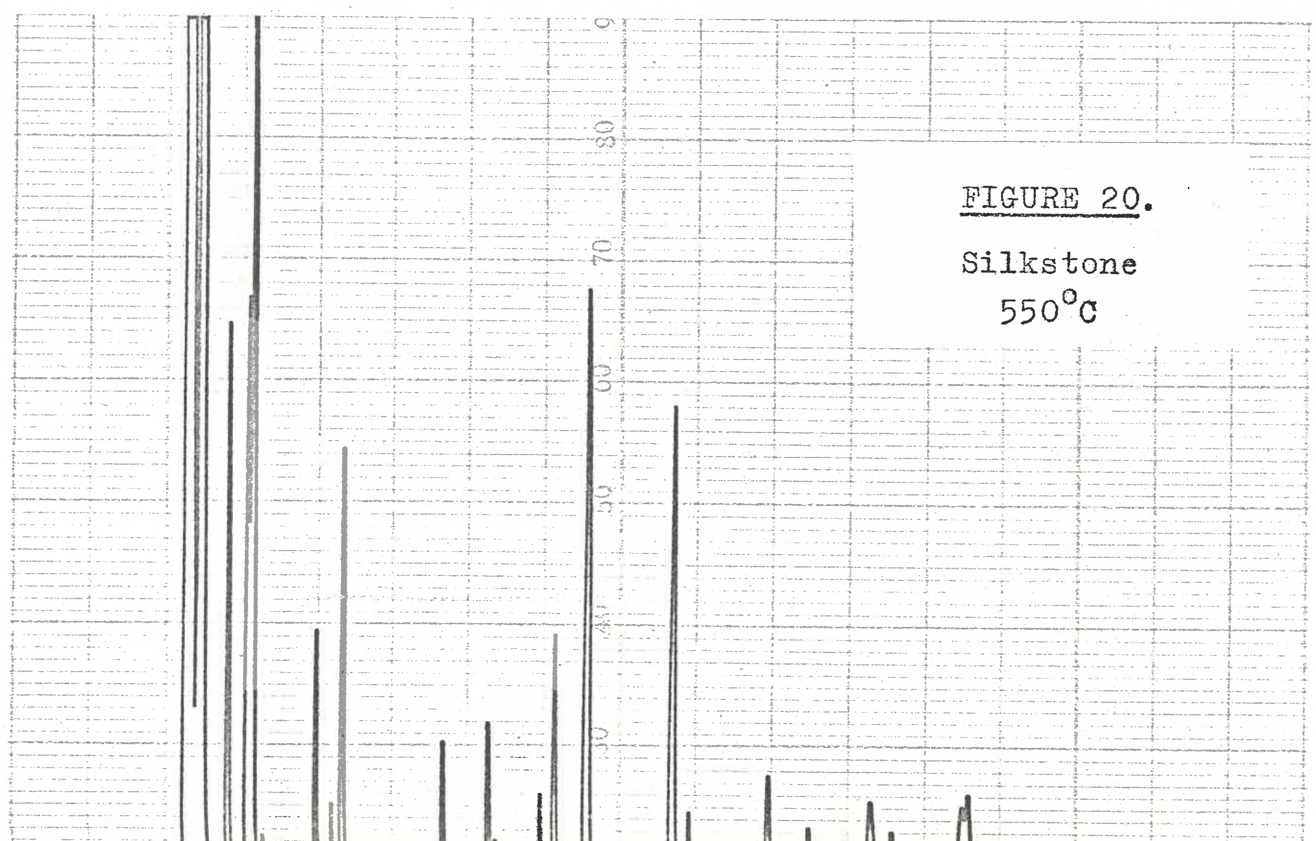
FIGURE 20.
Silkstone
550°C

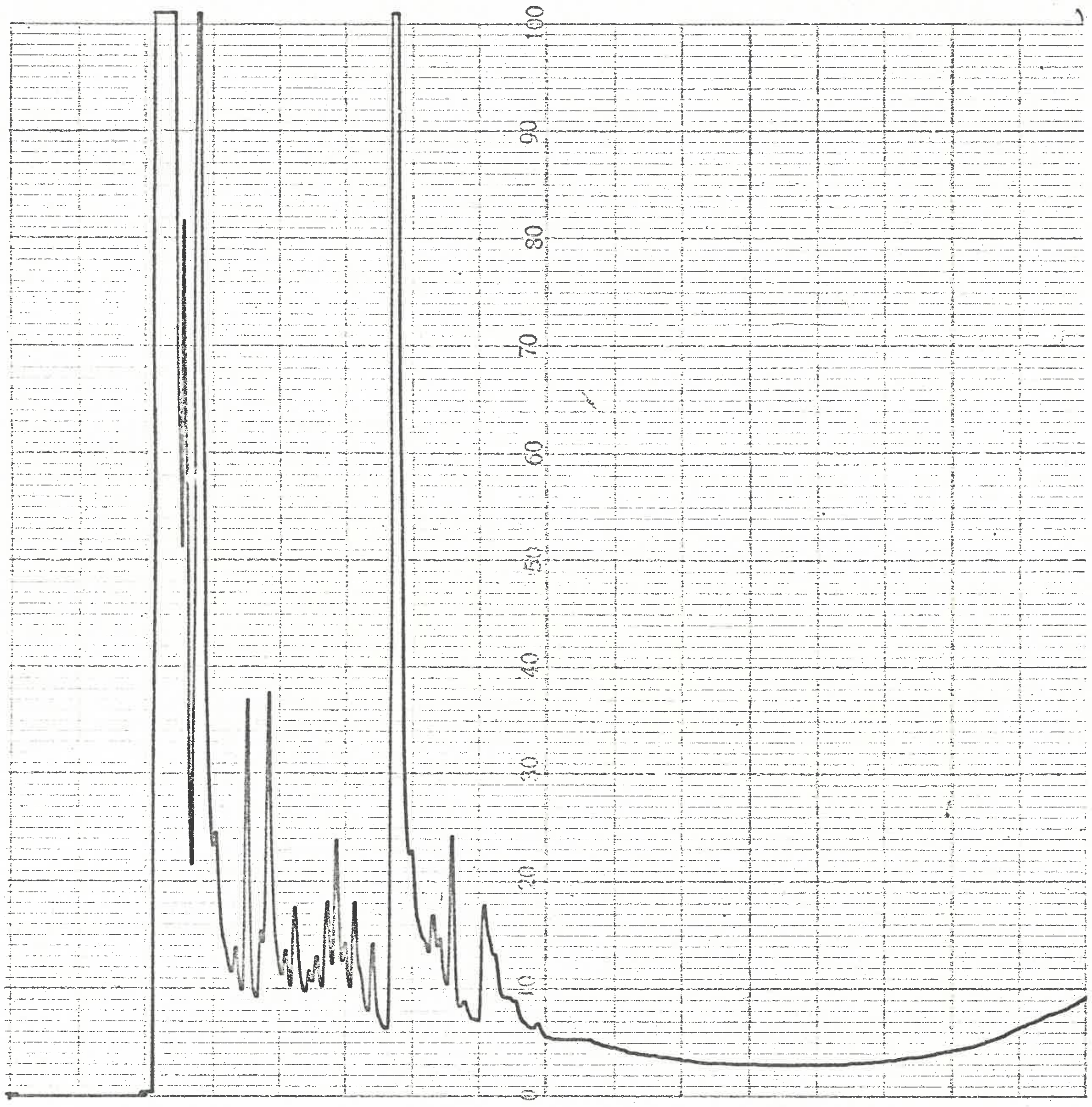

FIGURE 21. Gas chromatogram of the gases formed by a coal dust explosion in the Hartmann bomb.