## Report 9. Interrogation of Herr Clar

Subject: Synthetic Lubricating Oils.

Location and Date: Nienburg, May 13, 1945.

Interrogation conducted by: Major D. Morten, British, Ministry of Fuel and Power.

Reported by: Major D. Morten.

### Introduction

K. Clar is aged 58. Although seven names had been given in the Assessor's report as having arrived at Nienburg, five of these were only "Laborants", so apart from Velde, Clar was the only other one examined.

He was also Manager of one of the Ruhrchemie research laboratories dealing with the synthesis of lubricating oils including pilot plant and production. This work was carried out in connection with the German Air Ministry.

### GENERAL:

The synthesis was made from quite a wide range of olefines, of a boiling range 60°/200°C. equivalent to fractions from $C_6$ to $C_{13}$. These could be obtained from cracked Fischer-Tropsch spirit, or from the primary products from either the cobalt or iron catalyst, the whole thing depending entirely on the olefin content, which could be up to 70%. The primary olefines were the best, but others could also be used. It was essential to purify them considerably before the polymerisation process, and this purification consisted of washing with caustic potash to remove any alcohols and a vapour-phase treatment over alumina to remove all oxy compounds.

Polymerisation took place by simple agitation in contact with $AlCl_3$, using as low a temperature as possible to get reasonable reaction time. The best yield of high viscosity oil was then obtained at about 15°C., and a 24-hour reaction time using 100 parts of olefines and 6 parts of $AlCl_3$. Using this type of olefine feed the aluminium chloride goes fluid and forms a bottom layer known as contact oil. Clar had found that certain selected olefines, such as heptene,

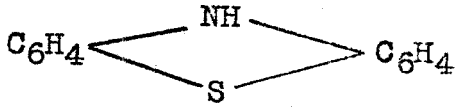
$C_7H_{14}$, gave no contact oil and produced an excellent lubricating oil of viscosity 8 to 10°E. at 50°C., with a V.I. of 125/130, but the conditions had been modified, at least in the laboratory, to give a bright stock of viscosity 50°E. at 50°C. Work on the selection of the best feed would have continued at Nienburg. Aluminium chloride can be added to the contact oil as necessary, and the temperature increased as the material loses its activity. The top layer contains the unchanged paraffins of gasoline boiling range, as well as the lubricating oil, and before distillation this was given a treatment of 3 hours at 170°/200°C. in an autoclave to reduce the chlorine content, using refining earth, MgO, ZnO, or zinc metal. The finished lubricating oils were separated by the usual distillation process. Although the oils were of excellent viscosity index, they were unstable to the action of heat and oxygen, and one of the main difficulties was to improve these properties against the B.A.M. oxidation test, or a modification developed by Clar. The details of this modification were to pass 15 litres of pure oxygen per hour through 175 grams of oil at 140°C. for 24 hours. No catalyst was used and the test does not appear sufficiently drastic. Various methods of treating the oils were tried :-

(1) Earth contacting at 200°C. This gave no success.

(2) Further heating with aluminium chloride using about 2% for 3 hours at 170°/200°C. This was found to improve the stability considerably, and gave fairly satisfactory results on the large scale.

(3) Various ways were being developed of adding sulphur to the oil as a stabiliser, a remarkable point being that this was added <u>before</u> the synthesis stage, and not in any way as a normal inhibitor to the finished oil.

The latest approved oil was made using 0.2/0.5% of phenthiazin. This material was made by heating diphenylamine and sulphur in the molecular proportion of one of the former to two of the latter; these were melted together and the temperature raised to 150°C. to react. The formula is :-



Two other "inhibitors", $\beta$-thionaphthol and its anthracene analogue, have also been used in this way, but laboratory results had indicated that ordinary flowers of sulphur was superior to sulphur in the combined form. This sulphur was added to the extent of 0.1/0.2% before the polymerising process, a second treatment with aluminium chloride being necessary to remove the smell from the first stage products. This oil was said to be oxygen-proof by Clar's method of test, and gave a viscosity change of only 10/20%, but the process had not been tried out on a large scale.

It was stated that the sulphur content of the finished oil was only a trace by analysis, and that the oil was non-corrosive to copper test. This oil had not yet been approved by the German Air Ministry, and Clar said that it was quite a new development.

The total aluminium chloride consumption is about 4% and one of the still outstanding problems was to dispose of the contact oil/spent catalyst mixture. This did not seem very difficult, as the oil could be separated merely by adding water.

As mentioned in the report on Velde's work, Clar's work was only on fractions boilding to 200°C., and he knew nothing of the work on the 300°/400°C. boiling range olefines.

The aluminium chloride catalyst does not need to be particularly pure, and can contain iron. In fact, for motor oils of 5°/8°E. viscosity it is possible to use mixed catalysts consisting of the chlorides of bismuth, iron and lead, but experiments on this were not completed. Molecular distillation of the lubricating oils in the laboratory gave fractions of about 2°E. viscosity with pour points down to -70°C., but normal fractionation on the large scale would give pour points down to -40°C.