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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

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I. G. FARBENINDUSTRIE - HOECHST GERMANY

Reported by

Lt. R. J. OZOL, U.S. Ord. Capt. C. C. CHAFFEE, U.S. Ord.

9 July 1945

CIOS Target No. 22/1g Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE G-2 Division, SHAEF (Rear) APO 413

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PERSONNEL OF TEAM (PART OF CIOS PARTY 551)

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I. INTRODUCTION

The offices of I.G. Farben, Hoechst, CIOS No.22/1g, Target of Opportunity for Group 30, were visited by a detachment from CIOS party 551, 9 July 45, to obtain further information on synthetic additives used in cutting and metal drawing oils, by interrogation of Dr. Hans Lange, Manager of the Dyestuff dept. who had worked on these materials.

II. MANUFACTURE OF EMULPHORS STH AND STX (HOECHST)

A. Preparation of Raw Material (Mersol).

The starting material was KOGASIN II, a special cut from the Fischer Tropsch synthesis having a distillation range of 220 - 330°C and having an average chain length of 15 carbon atoms. This material was first hydrogenated over a nickel tungstate catalyst then redistilled in order to remove olfinic and ocygenated compounds. The product resulting from these operations was termed MEPASIN and was then converted into the sulfone chloride derivitive by reaction with SO2 and chlorine in the presence of artificial light, according to the Reed process. This reaction was carried out at 30°C to obtain a maximum addition of S02Cl at the end of the hydrocarbon chain and a minimum substitution of chlorine elsewhere on the chain. This type of reaction was further assured by allowing the reaction to proceed only to approximately 50% completion. This product containing 50% unreacted oil was termed MERSOL and was normally reacted with caustic sode to make detergents.

B. Preparation of Emulphors.

A variety of emulsifying agents were made by reacting MERSOL with Ammonia to make an amide, which was reacted with monochloracetic acid before saponification with NaOH:

R-SO2NH2 / C1CH2COOH ---- R-SO2-NH-CH2COOH

R-SO₂-NH-CH₂COOH / ----- R-SO₂-NH-CH₂-COONs -

The pH was adjusted so that the emulsified water

solution was slightly above the neutral point. The commercial product contained 30-50% of the Na salt with 70-50% of mineral oil, MEPASIN. These compounds were marked as Emulphor STH and STX containing 50 and 30% mineral oil respectively.

III. PROPERTIES AND USES OF EMULPHORS STH AND STX.

For metal drilling and light cutting operations 1-1% of the emulsion of STH (Borhrmittel H) was stated to be as effective as a 5-10% emulsion of fatty acid soaps and oil previously used. Since soaps were in very short supply, 500 tons per month of Bohrmittel H was being manufactured at the end of the war. For heavy cutting 60% of the compound is mixed with 40% of mineral oil (3-50E, preferably unrefined) and an intended production of 600 tons per month was contemplated at Hoechst. Several small purchasers were already using this type of blend. For rust prevention in gasoline containers they used 0.1% by weight of Bohrmittel H in the gasoline. This material was effective for the purpose but as expected aggrevated engine deposits.

Since it was desired to combine the advantages of rust prevention and the injection of a small amount of water into the engines, further work was in progress to develop products in which the inorganic radicals were eliminated. Among some 200 products tested for the dual purpose, two proved most promising which are represented by the following formula.

$$c_{12H_{25}} - c_{6H_4} - 0 - NH_4$$

The first compound was obtained by the condensation of the sodium salt of dodecylphenyl with chloracetic acid and neutralization of the product with ammonia to form a salt. The second compound was formed by neutralizing iso-butyl-cyclohexyl-n-butyric acid with ammonia. The first compound was considered most promising, but test data from field trials was not available.

IV. USE OF METAL DRAWING OIL - "SAURE E".

"Saure E" is MEPASIN sulfamido acetic acid which is obtained by acetone extraction of the unreacted MEPASIN from Bohrmittel H. Plant scale tests have been made on the drawing of bonderized metals, starting with metal discs. Full-scale production tests were also made on the final draw of 3.7 cm. steel shells. The tests were only partly successful and no definite conclusion on the characteristics of the material can be made at present. The difficulties encountered when working with "Saure E" included the following:-

- a) Material treated with "Saure E" must be drawn immediately since the effective film tends to shrink, thereby leaving some metal areas uncovered;
- b) The solution of "Saure E" causes reddening and peeling of the skin.

According to the limited experience obtained, it seemed possible to overcome these difficulties by adjusting the pH of the working solution to values between 5 and 7. This was done by the addition of a zincate solution (sufficient to neutralize one-half of the "Saure E" used) to a 0.5% solution of "Saure E".

Better results, without the above-mentioned disadvantages, are expected from the use of isopropyl-cyclohexylbutyric acid which can be used in form of its Na salt and in exactly the same way as the ordinary scap. No production scale tests on this material have been made but laboratory tests indicate that it is about 5 times as efficient (calculated from the amount of chemical required for covering the metal surface) as the scap solution.

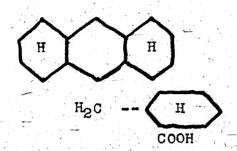
V. RESEARCH TRENDS IN SYNTHETIC EMULSIFYING AGENTS.

The raw material for the emulsifying agents was KOGASIN II obtained from the Fischer Tropsch synthesis. In order to be less dependent upon this source of material. a research program was inaugurated and consisted of condensing alkyl benzene or anthracene with phthalic anhyride in the presence of AlCl3 and then hydrogentating this type of compound. The chemical structure of the compound from alkyl benzene was of

the type

$$R - H - CH$$
 Where R is C_3 -

The compound from anthracene had the formula.



Laboratory scale work on the preparation of these compounds was underway and no conclusions may be made as to the additive properties.

VI. INFORMATION CONCERNING THE INTRODUCTION OF THE

A document from Hamburg, dated 5/2/44 issued by the Technical Advisory Committee for the Reich Station for Mineral Oils, entitled "Concerning the Introduction of the New Boring Medium "Ho", gives full information concerning the introduction and mode of use of the compound. A copy of this article is appended.

VII. SAMPLES.

A drum sample of Bohrmittel H has been obtained for test purposes either as a cutting oil, as a gasoline additive, or as a metal drawing oil in the extracted acid form.

18 July 1945.

APPENDIX A

Instructions for use of "Ho"

APPENDIX "A"

Hamburg 5.2.44.

TECHNISCHE GUTACHTER - KOMMISSION BEI DER REICHSSTELLE FUR MINERALOEL.

(Technical Advisory Committee for the Reich Station for Mineral Oils).

CONCERNING THE INTRODUCTION OF THE NEW BORING MEDIUM "Ho" .

All previous communications concerning introduction and mode of use are hereby superseded.

The supply position in the mineral oil industry renders it necessary to exert the utmost economy in the use of fats and mineral oils in metal-working. As a result of careful research in preparation and application, a new boring medium has been developed that contains no fats and allows of considerable saving in mineral oil.

Not only does this new product replace earlier boring oils, but it shows a whole series of favourable properties of considerable importance under present working conditions.

The boring medium "Ho" is made by the I.G.Dye Industry and has been in use for many months in several works. The amount of fats and mineral oils that can be saved in this way justifies the wide-spread introduction of the new medium.

The boring medium "Ho" has the following special properties.-

1. It can be used, without treatment, with mains water up to 20°DH. In the case of harder water the appropriate use of soda or permutit for softening is indicated, or condenser water may be used. Excess soda should be avoided; for 20°DH 0.4 gm./lt., for 30°DH 0.6 and for 40°DH, 0.8 gm. of soda ash is sufficient (after addition allow to stand a few hours, with occasional stirring). The water hardness can, if necessary, be obtained from the responsible water works.

To obtain as stable an emuision as possible, boring medium, "Ho" should first be agitated with 2-3 parts of cold water (base solution). The water should be added gradually, i.e., each portion added only when the preceding one has been well mixed in. This base solution can then be diluted in one operation with the main bulk of water, either by addition of the latter or by pouring into water. The final emulsion should not be very milky but translucent (opalescent)

The common salt content of most water supplies is below 200 mg./litre and is thus without any harmful corresive action. Salt concentrations above 500 mg./litre render the water unsuitable for emulsification, the rust-protection of the emulsions then falls sharply with increasing salt content. In such cases a suitable water supply, among others condenser water must be used. The appropriate water-works will also give information as regards NaCl content.

2. In general the addition of special protectives against corrosion is unnecessary, since boring medium "Ho" protects tool, job and machine against rust. However, if rust appears, the supplier and the Lubricants-Society should be informed. Often the addition of 1-2% soda-ash helps (calculated in the weight of boring medium Ho, i.e., add 10-20 gm. soda-ash per kilogram and stir well and only then dilute with water, as described above. The soda-ash is thus not added to the water or to the finished emulsion). Protectives against corrosion such as sodium nitrite etc., can be used, but, as with soda, excess should be avoided, since otherwise the essential stability of the emulsion is endangered.

The mixing ratio must therefore be kept much lower than with previous boring oils. On the average, for all operations previously undertaken with aqueous emulsions the ration 1.100 should not be exceeded. For grinding work 1.150 to 1.200 is sufficient. For tough material the ratio can be increased to 1.50 if necessary, when shayings are being taken off (zerspanen). Higher concentrations and increased soda addition cause strong foaming of the cooling liquid.

4. The boring medium "Ho" emulsion alters during use only when it becomes unstable, because of too hard water of excessive additives. It then runs off on the work or cuttings and the emulsion becomes thinned. In addition, It may become action after lengthy circulation. It is useful to test the reaction of the emulsion with a suitable

indicator (see below) from time to time and when necessary, to render it weakly alkaline by the slow addition of 1:10 soda solution. A pH of about 8 should be maintained, and under no circumstances should the neutral point (pH = 7) be passed. April of the blight vito according to the passed and the point of the blight of the blight of the second second bedoes a constant.

suitable as a lubricant. On the contrary, it washes away lubricating oil from surfaces gliding in contact, so that it cannot be used to replace cutting of with automatic machines. The lubricating oil collecting of the surface in the cooling liquid sump should be removed from time to time and after settling can be used for lubricating railway-beds etc. With shavings of grey cast pig iron separation of the boring medium emulsion sometimes occurs In this case a considerable addition of rust protective must be made, on in case of need another boring oil can be used.

Before using with the new boring medium emulsion, all deposits should be removed from the machines as far as is possible. An intermixing of the boring oil previously used with the new product should not be made, but the emulsions can be mixed.

The immediate introduction and appropriate use of the new product is essential from the point of view of war economy. The change over to the new medium should be so arranged that while the previous stock of boring oil is being used up, no confusion or mixing is possible.

Estimation of the Concentration of Boring Medium Hb and of the pH value.

The estimation of the concentration of boring medium "Ho" can be carried out as for boring oils. A boring oil tester can be conveniently used for the purpose.

The procedure is as follows .-

The flask of the boring oil tester is filled up to the mark with 100 c.cm. of the Hb emulsion to be tested. Then 30 c.cm. of a solution of 100 g. conc. sulphuric acid and 100 g. of common salt in 1000 c.cm. of water is added, up to the upper filling mark. The use of common salt is advantageous for obtaining better separation of the boring medium from the emulsion which is spilt up by the acid. Separation is also helped by standing the flask in a water bath at 60 - 80°C. Splitting up and separation are complete after about 30 minutes. This can be seen from the clearness of the liquid. The flask should now be cooled.

If ofly liquid still clings to the walls, aggregation can be effected by repeated rotations. The quantity of oily liquid separating is read off on the calibrated neck of the flask.

The number so obtained gives the quantity of "Ho" present in the 100 c.cm. tested and thus gives directly the percentage content of the emulsion. The estimation of the ph value can be made with Lyphan paper or with Merck's reagent paper.

Technical Advisory Committee

of the Covernment Station for Mineral Oil.

Sgd . E. Thiessen.

N.B. Differences from the communication of 11.10. 43 are shown by vertical margin lines.