Lubricating Oils

The normal oils prepared by Ruhrchemie would not pass the B.A.M. oxidation test, but were perfectly satisfactory in ordinary motor-car engines. The new oils prepared with the incorporation of sulphur or phenthiazine had not been subjected to the B.A.M. test, and no data were available to correlate this test and the German oxidation test, or either test with engine performance. The Luftwaffe had reported favourably on a 100 ton batch of the phenthiazine oil sent to them by Ruhrchemie.

The OXO Synthesis

Dr. Landgraf of Ruhrchemie was asked for data on the catalyst consumption in this process. He stated that for every 100 kg. of raw material with 35% olefines, 1 kg. of cobalt (i.e. 3 kg. of catalyst) was added. The catalyst could be re-used 50 to 100 times before regeneration in the catalyst factory was necessary and hence each kg. of cobalt would treat 5,000 to 10,000 kg. of raw material corresponding to a production of 1,700 to 3,300 kg. of alcohols.

The exothermic heat of reaction for the lower olefines was given as 35 to 45 kg.cal. per mole olefine. It was not necessary to take exceptional precautions in respect to temperature control when starting the reaction, but it was usual when treating Fischer-Tropsch olefines to start at 120 to 125°C. and raise the temperature finally to 135 - 140°C. For petroleum oils these temperatures should be increased by

10 to 20°C.

With a C_{12} α-olefine, about half the product should be straight-chain and half α-methyl branched, but in the presence of cobalt carbonyl about 5% of isomerisation (double-bond migration) occurs and the products are 45% normal and 55% iso.

Henkel have carried out experimental work on the OXO synthesis using olefines obtained by cracking petroleum slack wax. The main disadvantage of this raw material was the presence of sulphur which survived the cracking process and led to poisoning of the cobalt OXO catalyst. Thus, with cracked petroleum olefines, the catalyst could only be re-used 5 times instead of the 50 - 100 times possible with Fischer-Tropsch olefines. They are proposing to carry out trials at higher temperatures hoping to minimise the effect of sulphur.

The alcohols obtained from the cracked petroleum olefines gave quite satisfactory detergents.

Buhrchemie found that the OXO aldehydes obtained from C_6 - C_{10} olefines can be converted into esters by a modified Cannizzaro reaction followed by esterification in an acid medium. These esters have a low viscosity (1.2°E. at 50°C.) but extremely good viscosity temperature properties (Pole height 0.7 to 0.9) and a low setting point (below - 50°C.), and would find use as additives for lubricating oils. The low setting point is due to the presence of branched-chain isomers; the corresponding straight chain esters have a setting point of + 10°C. The following example of the preparation of the esters was given:-

As starting material crude aldehydes obtained by the OXO reaction from the 40 - 150°C. fraction of cracked spirit were used, and 2 kg. were heated for one hour at 150°C. with 100 g. of caustic soda in a closed vessel. After cooling, the yellowish product was treated with 10% sulphuric acid and the acidified mixture boiled under reflux for 20 hours. The mixture was then distilled to 225°C. (normal pressure) and the residue washed with 2% caustic soda and water. The yield of esters was 1,210 g.

The properties of the materials at various stages are given in Table 58.

Table 58.

Preparation of Ester Oils. Ruhrchemie

	Crude Aldehydes	Product after alkali treatment and acidification	Esters
d_{20}	0.810	-	0.870
Acid No.	3	110	0
Sap. No.	3	119	138
Hydroxyl No.	5	119	5
Carbonyl No. mg.CO/g.	140	5	1
n_d^{20}	-	-	1.4500
Setting point	-	-	-65°C.