

RESTRICTED

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ENCLOSURE (B) 24

EXPERIMENTAL METHOD FOR
MANUFACTURING ADDITIVE AGENTS

By

NAV. CHEM. ENG. N. MATSUO
CHEM. ENG. LT. COMDR. K. HIRATA

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ENCLOSURE (B)24

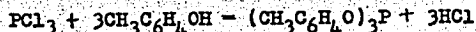
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ENCLOSURE (B)24

I. TRICRESYL PHOSPHITE

Phosphorous trichloride (30% wt.) is slowly added to cresol (70% wt.) with sufficient stirring at room temperature. The condensation reaction shown below takes place:



The resulting product is then slowly heated to about 200-250°C, leading inert gas (carbon dioxide or nitrogen) through the medium, in order to remove hydrogen chloride gas and to complete the condensation reaction. The product is finally fractionated in vacuum, and the fraction boiling from 223°C to 227°C at 5mm Hg is taken.

The process is schematically shown in Figure 1(B)24.

| | |
|---------------------------------|---------|
| Density (d_4^{24}) | 1.1283 |
| Ref. Index (n_D^{25}) | 1.5695 |
| B.P. (°C/10mm Hg) | 240-243 |

II. TRICRESYL PHOSPHATE

Phosphorous oxychloride (33% wt.) is slowly added to cresol (67% wt.) at room temperature. Anhydrous aluminium chloride (2-5% wt. of the mixture) is added little by little, and then heated to about 130-150°C, and maintained at that temperature for 7 hrs. A condensation reaction takes place according to the following equation.

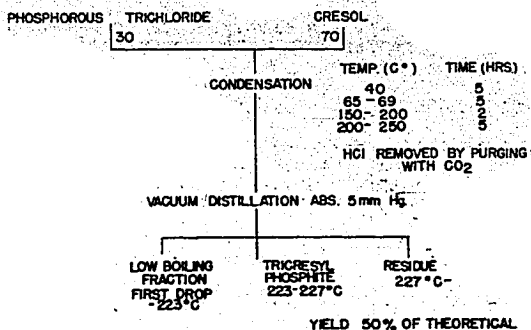


The reacted mixture is washed with water and, after being dried by heating, is fractionated in vacuum of 5mm Hg. The fraction boiling from 240°C to 250°C at 5mm Hg pressure is taken.

The process is schematically shown in Figure 2(B)24.

| | |
|---------------------------------|---------|
| Density (d_4^{20}) | 1.1718 |
| Ref. Index (n_D^{25}) | 1.5010 |
| B.P. (°C/760mm Hg) | 425-435 |

III. A detailed flow sheet of the pilot plant for production of additives is shown in Figure 3(B)24.



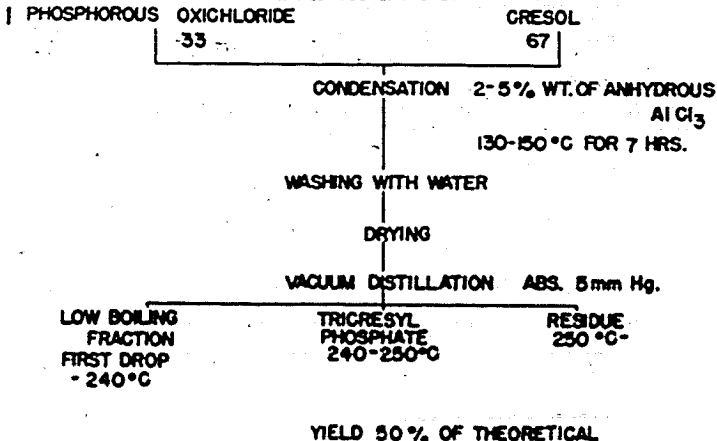
AVERAGE PROPERTIES OF THE PRODUCT

DENSITY d_4^{24} 1.283 B.P 240-243°C/10mm Hg
 REF. INDEX n_D^{25} 1.5695

Figure 1(B)24

PROCESS FLOW SHEET FOR

MANUFACTURING TRICRESYL PHOSPHATE



AVERAGE PROPERTIES OF THE PRODUCT

d_4^{20} 1.1716
 n_D^{25} 1.5010
 B.P. = 425-435°C/760 mm Hg.

Figure 2(B)24

PROCESS FLOW SHEET FOR

MANUFACTURING TRICRESYL PHOSPHATE

ENCLOSURE (B)24

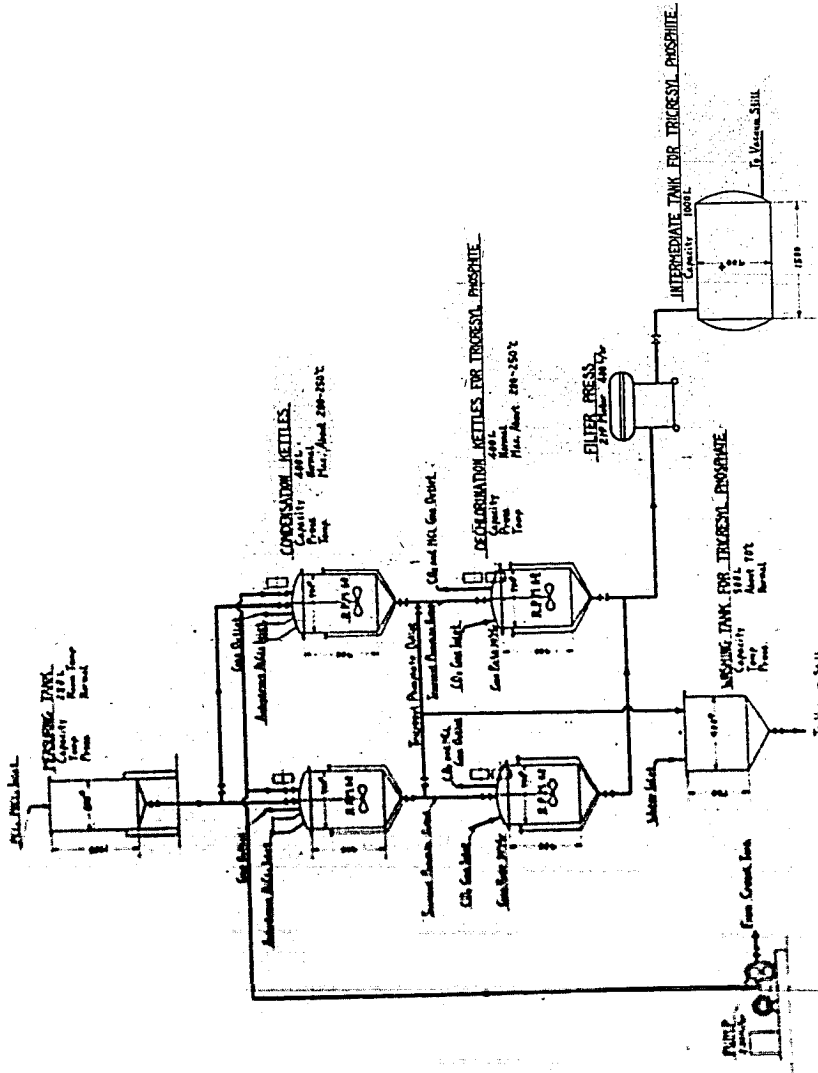


Figure 3(B)24
FLOW SHEET OF PILOT PLANT OF ADDITIVE AGENTS

ENCLOSURE (b) (2)

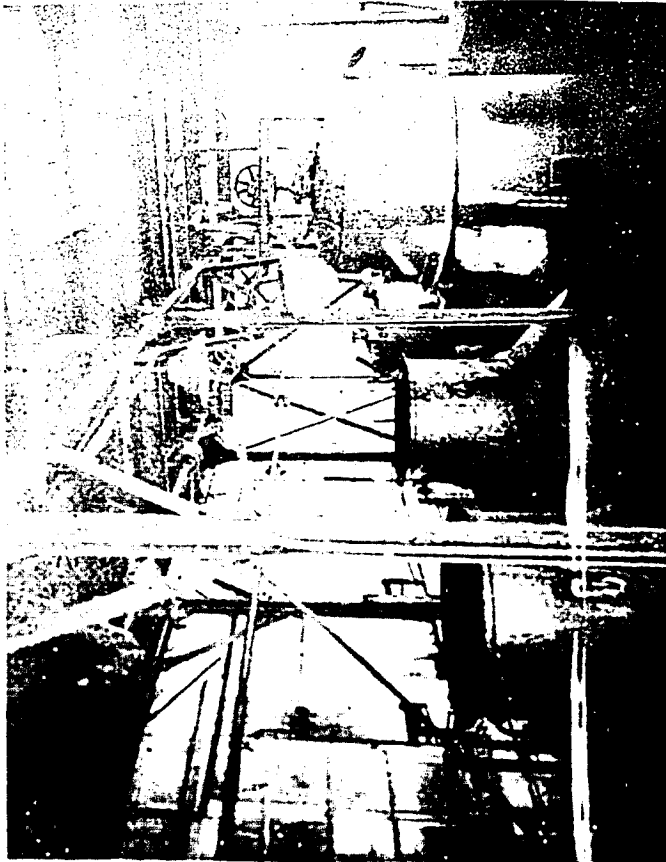


Figure 4(B)34
PILOT PLANT FOR MANUFACTURING
ADDITIVE AGENT OF LUBRICANT