

KOPPERS COMPANY, INC.





PLASTICS DIVISION KOPPERS TEST METHOD No. C-208

EVALUATION OF MOTOR BENZOL AND CRUDE LIGHT OIL

OCTOBER 9, 1956

SCOPES

THIS METHOD IS APPLICABLE TO ALL MOTOR BENZOLS AND ALL CRUDE LIGHT OILS EXCEPT WHERE CONTRACTURAL REQUIREMENTS SPECIFY A DIFFERENT PARTICULAR METHOD.

PRINCIPLES

THE EVALUATION OF MOTOR BENZOL AND CRUDE LIGHT OIL INVOLVES A PROCEDURE SIMILAR TO THAT USED IN COMMERCIAL PROCESSING. THE MATERIAL IS "TOPPED" TO REMOVE NON-CONDENSABLE SUBSTANCES AND "LIGHT ENDS." THE REMAINDER IS ACID WASHED, NEUTRALIZED, AND THEN DISTILLED TO RECOVER THE "PURE FRACTIONS" AND INTERMEDIATES.

APPARATUS:

- 1. PYREX, PEAR-SHAPED, 2000-ML. SEPARATORY FUNNEL WITH \$ STOPCOCK AND \$ STOPPER FISHER SCIENTIFIC COMPANY, CAT. No. 10-437.
- 2. Pyrex graduated cylinders:

 - D. 250-ML. CAPACITY; 2-ML. SUBDIVISIONS 00 00 E. 500-ML. CAPACITY; 5-ML. SUBDIVISIONS 00 00
 - F. 1000-ML. CAPACITY; 10-ML. SUBDIVISIONS 8-553
- 3. Pyrex Funnel, 150-mm. DIAMETER AND 150-mm. STEM LENGTH FISHER SCIENTIFIC COMPANY, CATALOG No. 10-372.
- 4. A. SECONDARY DISTILLATION THERMOMETER, -5°C TO 250°C, FULL LENGTH, INTERCHANGEABLE 10/30 \$ GROUND JOINT ARTHUR H. THOMAS Co., CATALOG No. 9531-C.
 - B. Specific gravity thermometer, -20°C to 105°C, etched stem, 12° Long Taylor Instrument Company, Catalog No. 21250.
 - C. LIGHT ENDS THERMOMETER, 72°C TO 126°C, 0.2°C DIVISIONS PRINCO CATALOG NO. 0235.
 - D. POT AND GOLUMN THERMOMETER, -2°C TO 300°C, 1°C DIVISIONS PRINGO CATALOG No. 065.

- 5. Distillation column 53" overall length x 1" diameter (1.D.) glass column with 35/20 spherical joint at the bottom and a 29/42 standard taper joint at the top. Nichrome wire coil on inner jacket provides heat for column. The packed length of the column is 48"; 44" are packed with Heli-Pak or 3/32" stainless steel helices and 4" are packed with 1/4" Berl Saddles.
- 6. HELIGRID PACKING "HELI-PAK" PODBIELNIAK, INC., CHICAGO, ILL., CATALOG No. 2917; OR STAINLESS STEEL HELIGES, 3/32" GOIL DIAMETER, No. 30 GAUGE SCIENTIFIC GLASS APPARATUS Co., CAT. No. J-1219.
- 7. CORAD VARIABLE REFLUX STILL HEAD CORNING GLASS WORKS, CAT. No. 3530.
- 8. Single neck, round bottom distilling flasks, 35/20 semi-ball joint with 24/40 \$ joint at 20° angle:
 - A. 1-LITER GAPACITY SCIENTIFIC GLASS APPARATUS Co., CAT. No. SB-1850.
 - B. 2-LITER CAPACITY " " "
 - C. INNER CONNECTING GLASS JOINTS WITH REDUCED TUBES 24/40 \$ SCIENTIFIC GLASS APPARATUS CO., CAT. No. J-180.
- 9. SPHERICAL HEATING MANTLES:
 - A. I-LITER CAPACITY SCIENTIFIC GLASS APPARATUS Co., CAT. No. FH-108.
 B. 2-LITER CAPACITY " FH-110.
- 10. "VARIAGS" GENERAL RADIO COMPANY, TYPE V-5MT.
- 11. FREEZING POINT APPARATUS SEE REFERENCE (1).
- 12. COLD TRAP FOR NON-CONDENSABLES USE GRADUATED TEST TUBE (INLET TUBE ATTACHED WITH CORK AND RUBBER TUBING) IMMERSED IN DRY ICE-ACETONE BATH.
 TEST TUBE (25 ML.) OBTAINED FROM FISHER SCIENTIFIC CO., CAT. No. 14-950.

REAGENTS:

- 1. CONGENTRATED SULFURIC ACID; REAGENT GRADE (96 + 0.5% H2SO4 BY WT.).
- 2. CAUSTIC SODA SOLUTION 15-20% BY WT. REAGENT GRADE NAOH.
- 3. CALCIUM CHLORIDE (FLAKES; TECHNICAL GRADE).

PROCEDURE:

A ONE-LITER SAMPLE OF THE OIL AT ROOM TEMPERATURE IS MEASURED IN A ONE-LITER GRADUATED CYLINDER; NOTE AND RECORD TEMPERATURE. THE SAMPLE IS TRANSFERRED TO 2-LITER DISTILLING FLASK AND THE FLASK CONNECTED TO THE COLUMN. THE OUTLET TUBE FROM THE STILL HEAD IS CONNECTED TO A COLD TRAP COOLED TO -30 TO -40°C IN A DRY ICE-ACETONE BATH. HEAT IS APPLIED TO THE FLASK BY MEANS OF A "VARIAC" TRANSFORMER AND "GLASCOL" HEATING MANTLE (LOWER HEMISPHERE ONLY) AND THE MATER-IAL IS REFLUXED UNTIL A CONSTANT VAPOR TEMPERATURE IS ATTAINED. THE LOW BOILING CONSTITUENTS OF THE OIL ARE RECOVERED IN THE COLD TRAP AND THE VOLUME IS MEASURED AND RECORDED. THESE LOW BOILERS MAY BE WARMED TO THE POINT WHERE BOILING BEGINS BEFORE READING VOLUME IN THE GRADUATED COLD TRAP. A WATER-COOLED AUXILIARY

CONDENSER IS ATTACHED TO THE OUTLET FROM THE STILL HEAD; AND A 50-ML. GRADUATED CYLINDER, IMMERSED IN A BEAKER OF ICE AND WATER, IS THEN PLACED TO RECEIVE THE DISTILLATE FROM THE CONDENSER. THE DISTILLATE IS COLLECTED AT A 20:1 REFLUX RATIO UNTIL THE OVERHEAD VAPOR TEMPERATURE IS 79.0°C, CORRECTED TO 760 MM. HG. ABSOLUTE PRESSURE. (NOTE: ALL TEMPERATURES REFERRED TO ARE CORRECTED TEMPERATURES.) AT THIS POINT THE DISTILLATION IS INTERRUPTED AND THE TOTAL VOLUME OF DISTILLATE OBTAINED IS RECORDED.

AFTER ALLOWING THE COLUMN TO DRAIN FOR AT LEAST ONE HOUR (PREFERABLY TWO HOURS), THE FLASK IS DISCONNECTED AND THE CONTENTS POURED INTO A 1000-ML. GRADUATED CYLINDER, GOOLED TO THE SAME TEMPERATURE AT WHICH THE ORIGINAL SAMPLE WAS MEASURED; AND THE VOLUME IS THEN RECORDED. USING A 2-LITER SEPARATORY FUNNEL, THE OIL IS WASHED WITH A VOLUME OF CONCENTRATED SULFURIC ACID EQUAL TO 7% OF THE VOLUME OF THE TOPPED OIL. THE ACID IS ADDED IN INCREMENTS OF 3-5 ML., SHAKING VIGOROUSLY BETWEEN EACH ADDITION. THE SEPARATORY FUNNEL MUST BE COOLED UNDER TAP WATER OGGASIONALLY TO KEEP THE TEMPERATURE OF THE OIL BELOW 25°C. WHEN ALL THE ACID HAS BEEN ADDED, THE MIXTURE SHOULD BE SHAKEN VIGOROUSLY AND GONTINUOUSLY FOR 20 MINUTES. THE MIXTURE IS THEN ALLOWED TO SETTLE FOR AT LEAST 2 HOURS OR PREFERABLY OVERNIGHT TO INSURE GOOD SEPARATION OF THE ACID AND OIL LAYERS. THE ACID LAYER (I.E., LOWER LAYER) IS THEN DRAWN OFF AND DISCORDED. THE OIL LAYER IS ADJUSTED TO THE ORIGINAL TEMPERATURE AND THE VOLUME AGAIN MEASURED (IN A LITER GRADUATE) AND RECORDED.

THE ACID-WASHED OIL IS NEUTRALIZED IN A SEPARATORY FUNNEL BY AGITATING FOR AT LEAST 5 MINUTES WITH 100 ML. OF 15-20% NAOH SOLUTION. WHEN THE CAUSTIC LAYER (1.6., LOWER LAYER) HAS SEPARATED, IT IS WITHDRAWN AND DISCARDED. THE SAMPLE IS THEN THOROUGHLY DRIED BY PASSING THROUGH CALCIUM CHLORIDE CONTAINED IN FILTER PAPER (No. | WHATMAN, 24.0-cm.). A 500-ML. PORTION OF THE DRY OIL IS MEASURED AND TRANSFERRED TO A ONE-LITER DISTILLING FLASK. A 100-ML. PORTION OF CHASER (BOILING OVER 200°C) IS ADDED TO THE SAMPLE IN THE FLASK. (NOTE: POLYETHYL-BENZENE TOPPED TO 210°C OR TETRALIN IS SATISFACTORY.) THE MATERIAL IS THEN FRACTIONALLY DISTILLED AND VOLUME-TEMPERATURE DATA OBTAINED AT A SUFFICIENT NUMBER OF POINTS DURING THE DISTILLATION SO THAT AN ACCURATE DISTILLATION CURVE MAY BE PLOTTED. THE DISTILLATION IS CONDUCTED USING A 20: | REFLUX RATIO BETWEEN PLATEAUS AND A 5: | REFLUX RATIO WHILE "PURE COMPONENTS" (1.6. BENZENE, TOLUENE, XYLENE) ARE BEING DISTILLED. THE BENZENE FRACTION INCLUDES MATERIAL DISTILLING UP TO 95.0°C. A GUT IS TAKEN AT THIS POINT. WHEN ALL THE BENZENE HAS BEEN DISTILLED, THE MATERIAL COLLECTED BETWEEN 79.0°C AND 95.0°C IS COMBINED.

THE FREEZING POINT (1) AND THE PARAFFIN CONTENT (2) OF THIS MATERIAL ARE THEN DETERMINED.

THE PERCENTAGE OF TOLUENE PRESENT IS BASED ON THE AMOUNT COLLECTED BETWEEN 95.0°C AND 125.0°C. A TOLUENE FRACTION WITH A 1.0°C BOILING RANGE INCLUDING THE BOILING POINT OF TOLUENE (110.6°C) IS COLLECTED AND THE PARAFFIN CONTENT DETERMINED.

THE XYLENE CONTENT IS BASED ON THE VOLUME OBTAINED BETWEEN 125.0°C AND 155.0°C, WHILE THE MATERIAL BOILING BETWEEN 155.0°C AND 200.0°C IS DESIGNATED AS HEAVY SOLVENT. THE DISTILLATION IS CONTINUED UNTIL A VAPOR TEMPERATURE OF 200°C IS REACHED; THEN THE DISTILLATION IS STOPPED.

IN ORDER TO AVOID ERRORS DUE TO LOSSES DURING THE DISTILLATION, A MATERIAL BALANCE ON A VOLUME BASIS SHOULD BE MADE. CONSIDERING COLUMN HOLDUP (ABOUT. 18 ML. AS TETRALIN), THE LOSS SHOULD NOT BE MORE THAN 4% OF THE VOLUME CHARGED TO THE COLUMN FOR THE SECONDARY DISTILLATION.

CALGULATIONS:

1. PRIMARY DISTILLATION

- % LIGHT ENDS = VOLUME COLLECTED BELOW 79.0 C x 100
- B. % ACID WASH LOSS = (VOL. BEFORE WASH VOL. AFTER WASH) x (100-A) VOL. OF OIL WASHED

2. SECONDARY DISTILLATION

- G. CORRECTION FOR OIL REMOVED # 100 A B
- D. VOLUME OF SAMPLE EMPLOYED AS STILL CHARGE IN SECONDARY DISTILLATION
- E. % BENZENE = VOLUME HYDROCARBON COLLECTED BELOW 95.0°C x C
- F. % TOLUENE # VOLUME COLLECTED BETWEEN 95.0°C AND 125.0°C x C
- % XYLENES = VOLUME COLLECTED BETWEEN 125.0°C AND 155.0°C x C
- H. % HEAVY SOLVENT = Volume collected between 155.0°C and 200.0°C x C
- 1. % HIGH BOILERS 100 (A + B + E + F + G + H)

RESULTS TO BE REPORTED:

THE FOLLOWING RESULTS SHOULD BE REPORTED:

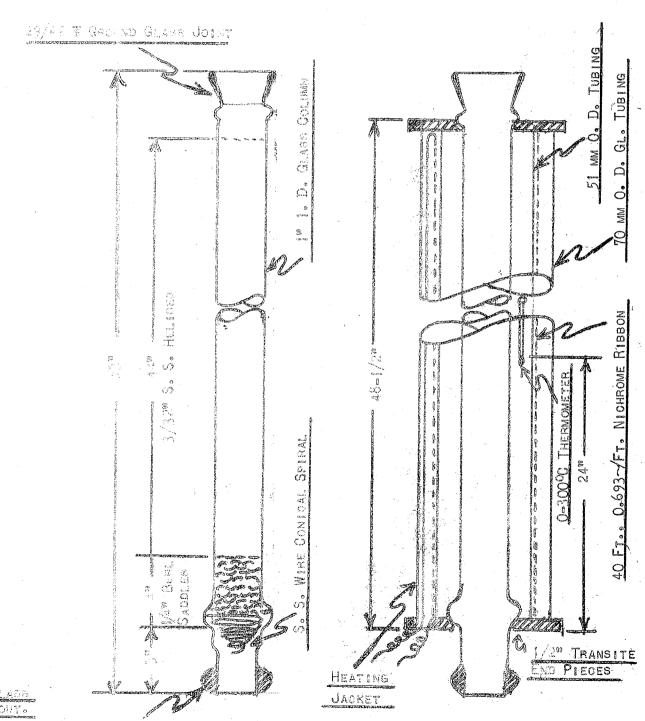
- % LIGHT ENDS
- % ACID WASH LOSS
- % BENZENE
- % TOLUENE
- % XYLENES
- % HEAVY SOLVENT % HIGH BOILERS
- FREEZING POINT OF THE BENZENE FRACTION DISTILLING BETWEEN 79.0 AND 95.000
- PARAFFIN CONTENT OF THE BENZENE FRACTION DISTILLING BETWEEN 79.0 AND 95.0°C.
- 10. PARAFFIN CONTENT OF THE TOLUENE FRACTION WITH A 10 BOILING RANGE, INCLUDING 110.6°C.

Items 8, 9, and 10 are not utilized in the calculations, but are used to indicate the quality of the separated components.

REFERENCES:

- (1) STANDARD METHOD OF TEST FOR SOLIDIFYING POINT OF BENZENE A.S.T.M. DESIGNATION: D-852-47.
- (2) STANDARD METHOD OF TEST FOR PARAFFINS IN INDUSTRIAL AROMATIC HYDRO-CARBONS - A.S.T.M. DESIGNATION: D-851-47.

DETAILS OF DISTILLATION COLUMN KARLESS TEXT METHOD No. C-208



Note: Pyrex Grace Throughour.

39/20 See 81082 John