



# KOPPERS COMPANY, INC.

CHEMICAL DIVISION  
 PITTSBURGH · 19 · PA.



## PLASTICS DIVISION KOPPERS TEST METHOD No. C-208

EVALUATION OF MOTOR BENZOL AND CRUDE LIGHT OIL

OCTOBER 9, 1956

### SCOPE:

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

### PRINCIPLE:

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  $\frac{3}{4}$  STOPCOCK AND  $\frac{3}{4}$  STOPPER - FISHER SCIENTIFIC COMPANY, CAT. No. 10-437.
2. PYREX GRADUATED CYLINDERS:
 

A.	10-ML. CAPACITY;	0.1-ML. SUBDIVISIONS	=	FISHER SCIENTIFIC COMPANY,	
				CATALOG No. 8-552	
B.	50-ML. CAPACITY;	1-ML. SUBDIVISIONS	=	"	"
C.	100-ML. CAPACITY;	1-ML. SUBDIVISIONS	=	"	"
D.	250-ML. CAPACITY;	2-ML. SUBDIVISIONS	=	"	"
E.	500-ML. CAPACITY;	5-ML. SUBDIVISIONS	=	"	"
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,  $-5^{\circ}\text{C}$  TO  $250^{\circ}\text{C}$ , FULL LENGTH, INTERCHANGEABLE 10/30  $\frac{3}{4}$  GROUND JOINT - ARTHUR H. THOMAS Co., CATALOG No. 9531-C.
  - B. SPECIFIC GRAVITY THERMOMETER,  $-20^{\circ}\text{C}$  TO  $105^{\circ}\text{C}$ , ETCHED STEM, 12" LONG - TAYLOR INSTRUMENT COMPANY, CATALOG No. 21250.
  - C. LIGHT ENDS THERMOMETER,  $72^{\circ}\text{C}$  TO  $126^{\circ}\text{C}$ , 0.2 $^{\circ}\text{C}$  DIVISIONS - PRINCO CATALOG No. 0235.
  - D. POT AND COLUMN THERMOMETER,  $-2^{\circ}\text{C}$  TO  $300^{\circ}\text{C}$ , 1 $^{\circ}\text{C}$  DIVISIONS - PRINCO CATALOG No. 065.



CONDENSER IS ATTACHED TO THE OUTLET FROM THE STILL HEAD; AND A 50-ML. GRADUATED CYLINDER, IMMERSSED 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  $79.0^{\circ}\text{C}$ , 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, COOLED 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 OCCASIONALLY TO KEEP THE TEMPERATURE OF THE OIL BELOW  $25^{\circ}\text{C}$ . WHEN ALL THE ACID HAS BEEN ADDED, THE MIXTURE SHOULD BE SHAKEN VIGOROUSLY AND CONTINUOUSLY 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 DISCARDED. 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 (I.E., 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. 1 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  $200^{\circ}\text{C}$ ) IS ADDED TO THE SAMPLE IN THE FLASK. (NOTE: POLYETHYL-BENZENE TOPPED TO  $210^{\circ}\text{C}$  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 20:1 REFLUX RATIO BETWEEN PLATEAUS AND A 5:1 REFLUX RATIO WHILE "PURE COMPONENTS" (I.E. BENZENE, TOLUENE, XYLENE) ARE BEING DISTILLED. THE BENZENE FRACTION INCLUDES MATERIAL DISTILLING UP TO  $95.0^{\circ}\text{C}$ . A CUT IS TAKEN AT THIS POINT. WHEN ALL THE BENZENE HAS BEEN DISTILLED, THE MATERIAL COLLECTED BETWEEN  $79.0^{\circ}\text{C}$  AND  $95.0^{\circ}\text{C}$  IS COMBINED.

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

THE PERCENTAGE OF TOLUENE PRESENT IS BASED ON THE AMOUNT COLLECTED BETWEEN  $95.0^{\circ}\text{C}$  AND  $125.0^{\circ}\text{C}$ . A TOLUENE FRACTION WITH A 1.0 C BOILING RANGE INCLUDING THE BOILING POINT OF TOLUENE ( $110.6^{\circ}\text{C}$ ) IS COLLECTED AND THE PARAFFIN CONTENT DETERMINED.<sup>(2)</sup> THE XYLENE CONTENT IS BASED ON THE VOLUME OBTAINED BETWEEN  $125.0^{\circ}\text{C}$  AND  $155.0^{\circ}\text{C}$ , WHILE THE MATERIAL BOILING BETWEEN  $155.0^{\circ}\text{C}$  AND  $200.0^{\circ}\text{C}$  IS DESIGNATED AS HEAVY SOLVENT. THE DISTILLATION IS CONTINUED UNTIL A VAPOR TEMPERATURE OF  $200^{\circ}\text{C}$  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.

**CALCULATIONS:**

1. PRIMARY DISTILLATION

A. % LIGHT ENDS =  $\frac{\text{VOLUME COLLECTED BELOW } 79.0 \text{ C} \times 100}{1000}$

B. % ACID WASH LOSS =  $\frac{(\text{VOL. BEFORE WASH} - \text{VOL. AFTER WASH}) \times (100-A)}{\text{VOL. OF OIL WASHED}}$

2. SECONDARY DISTILLATION

C. CORRECTION FOR OIL REMOVED =  $100 - A - B$

D. VOLUME OF SAMPLE EMPLOYED AS STILL CHARGE IN SECONDARY DISTILLATION

E. % BENZENE =  $\frac{\text{VOLUME HYDROCARBON COLLECTED BELOW } 95.0^{\circ}\text{C} \times C}{D}$

F. % TOLUENE =  $\frac{\text{VOLUME COLLECTED BETWEEN } 95.0^{\circ}\text{C AND } 125.0^{\circ}\text{C} \times C}{D}$

G. % XYLENES =  $\frac{\text{VOLUME COLLECTED BETWEEN } 125.0^{\circ}\text{C AND } 155.0^{\circ}\text{C} \times C}{D}$

H. % HEAVY SOLVENT =  $\frac{\text{VOLUME COLLECTED BETWEEN } 155.0^{\circ}\text{C AND } 200.0^{\circ}\text{C} \times C}{D}$

I. % HIGH BOILERS =  $100 - (A + B + E + F + G + H)$

RESULTS TO BE REPORTED:

THE FOLLOWING RESULTS SHOULD BE REPORTED:

1. % LIGHT ENDS
2. % ACID WASH LOSS
3. % BENZENE
4. % TOLUENE
5. % XYLENES
6. % HEAVY SOLVENT
7. % HIGH BOILERS
8. FREEZING POINT OF THE BENZENE FRACTION DISTILLING BETWEEN 79.0 AND 95.0°C.
9. 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.

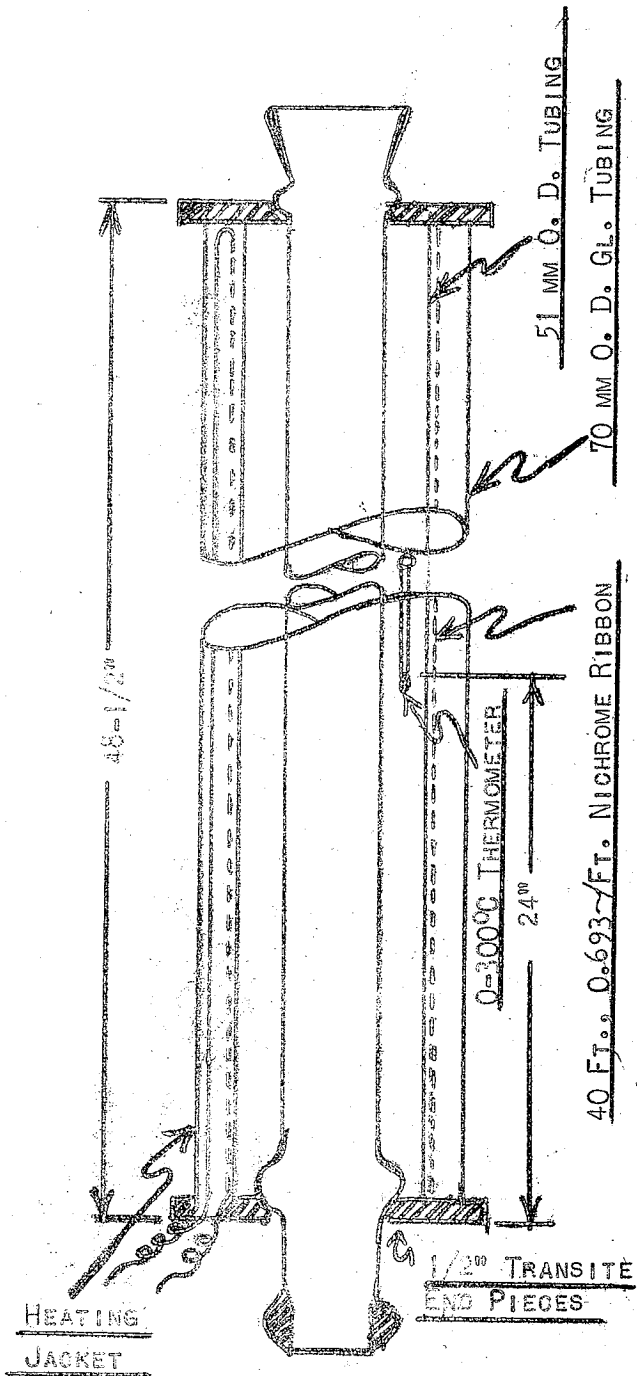
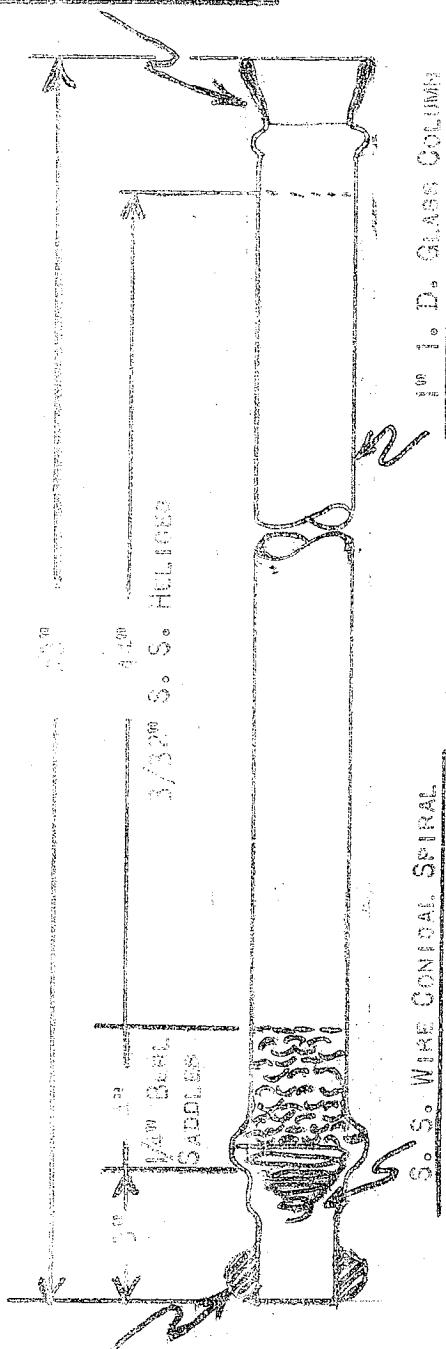
ITEMS 8, 9, AND 10 ARE NOT UTILIZED IN THE CALCULATIONS, BUT ARE USED TO INDICATE THE QUALITY OF THE SEPARATED COMPONENTS.

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 HYDROCARBONS - A.S.T.M. DESIGNATION: D-851-47.

DETAILS OF DISTILLATION COLUMN  
KOPPEL TEST METHOD No. C-208

29/42 T GROUND GLASS JOINT



NOTE: PYREX GLASS  
THROUGHOUT.

35/40 SFF-F1041 JOINT