ethyl caproate and 72.4 g. (89%) of caproic acid. The yield of caproic acid, based on unrecovered ester, was 90%. The gain in weight of the Ascarite tube was less than 0.1 g.

Pyrolysis of Ethyl Stearate at 600°. When 55.6 g. of ethyl stearate were pyrolyzed in the same apparatus at 600°, the pyrolysate was condensed in a series of five traps that were cooled in a dry ice–methyl cellosolve bath to obtain 34.9 g. of condensable material. Distillation of the crude liquid pyrolysate through a 12-in. Vigreux column yielded 10.9 g. (22%) of stearic acid, m. p. 59–63°, plus a large amount of low boiling liquids. The gain in weight of the Ascarite tube was less than 0.4 g.

The gaseous pyrolysate that did not condense in the dry ice traps was passed through a solution of 150 g. of bromine in 1,000 ml. of carbon tetrachloride. The carbon tetrachloride solution was washed with a 5% sodium sulfite solution until the brown color disappeared and then with 100 ml. of water. After it was dried over calcium chloride, the solution was distilled through a 10-in., helix-packed column to yield 60.2 g. of ethylene dibromide, b.p. 122–130°, n_D^{25} 1.5370 [reported (12) b.p. 130°, n_D^{25} 1.5379]. If one were to assume that each molecule of ethyl stearate would liberate only one molecule of ethylene, only 33.4 g. of ethylene dibromide would be expected.

Saponification of Ethyl Stearate. A mixture of 100 g. (0.32 mole) of ethyl stearate, 200 ml. of water, 1,300 ml. of ethyl alcohol, and 35.8 g. (0.64 mole) of potassium hydroxide was heated under reflux for 5 hrs. (although 30 min. may have been sufficient). After 500 ml. of water had been added and 1,200 ml. of ethyl alcohol had been removed by distillation, the residue was acidified with dilute sulfuric acid. The reaction mixture was then extracted with three 250-ml. portions of a benzene-ether mixture, and the combined extracts were dried over anhydrous magnesium sulfate. After the solvents were removed by evaporation, the residue was distilled through a 12-in. Vigreux column to yield 79.5 g. of stearic acid, b.p. 153–161° (0.02 mm.), m.p. 66.5–70°. The residue in the pot and column was recrystallized from a water-dioxane mixture to give an additional 4.7 g. of stearic acid, m.p. 68–69.5°. The total yield of stearic acid was 84.2 g. (92%).

Ethyl 2, 4, 6-Trimethylbenzoate. To a 1-liter, three-necked flask containing 9.19 g. of magnesium and 20 ml. of ether was added dropwise a solution of 75.6 g. (0.38 mole) of bromomesitylene in 100 ml. of anhydrous ether. After the reaction mixture had been heated under reflux for 24 hrs., approximately one-fourth of the magnesium remained unreacted. Over a period of 30 min. 70 g. (0.68 mole) of freshly distilled ethyl chloroformate were added to the Grignard solution. After the reaction mixture had been heated for an additional 24 hrs., dilute sulfuric acid was added until two layers separated. The aqueous layer was extracted with two 50-ml. portions of ether, and the combined ether layers were washed successively with 50 ml. of 1*N* sodium hydroxide, two 50-ml. portions of a 5% potassium bicarbonate solution, and two 50 ml. portions of water. The ether solution was dried over anhydrous magnesium sulfate, and the ether was removed by distillation. The residue was fractionated through a 10-in., helix-packed column to yield 35.7 g. (50%) of ethyl 2,4,6-trimethylbenzoate.

Pyrolysis of Ethyl 2, 4, 6-Trimethylbenzoate. When 10.4 g. of ethyl 2,4,6-trimethylbenzoate were pyrolyzed by the same procedure, considerable charring occurred. The brown pyrolysate was dissolved in 150 ml. of ether, and this ether solution was extracted with three 25-ml. portions of 1*N* sodium hydroxide. The combined extracts were cooled in an ice bath and acidified with concentrated sulfuric acid. The resulting solid was removed by filtration and dried over night under vacuum to yield 7.78 g. (88%) of 2, 4, 6-trimethylbenzoic acid.

REFERENCES

1. Bailey, W. J., and Golden, H. R., J. Am. Chem. Soc., *75*, 4780 (1953).
2. Bailey, W. J., and Hewitt, J. J., J. Org. Chem., *21*, 543 (1956).
3. Bailey, W. J., and Rosenberg, J., J. Am. Chem. Soc., *77*, 73 (1955).
4. Brown, H. C., and Kornblum, R. B., J. Am. Chem. Soc., *76*, 4514 (1954).
5. Fieser, L. F., and Fieser, M., "Organic Chemistry," 2nd ed., p. 416, Boston, Mass., D. C. Heath and Co., 1950; Markley, K. S., "Fatty Acids," p. 581, New York, N. Y., Interscience Publishers Inc., 1947.
6. Halbig, P. (Lonza Elektrizitätswerke und Chemische Fabriken A–G), U. S. 2,286,407 (1942).
7. Kraft, F., Ber., *16*, 1719 (1883).
8. Ladburg, J. E., and Turner, E. E., J. Chem. Soc., 3885 (1954).
9. Newman, M. S., J. Am. Chem. Soc., *63*, 2434 (1941).
10. Ratchford, W. P., "Organic Syntheses," vol. 29, p. 2, New York, N. Y., John Wiley and Sons Inc., 1949.
11. Ratchford, W. P., Rehberg, C. E., and Fisher, C. H., J. Am. Chem. Soc., *66*, 1864 (1944).
12. Shriner, R. L., and Fuson, R. C., "Systematic Identification of Organic Compounds." 2nd ed., p. 217, New York, N. Y., John Wiley and Sons Inc., 1940.

[Received January 13, 1956]

Report of Cellulose Yield Committee—1955-1956

GIVEN below are the checked yield analyses obtained on samples sent out during the year to 10 laboratories. During this period three samples were sent out three times during the season. Two samples were made up of second-cut linters and one hull fiber.

Very good checked analyses were obtained, as shown by the above table. It is recommended that this work be continued, that is, samples sent out at least three times a year to all laboratories equipped to run cellulose yield analyses.

Lab. No.	No. of Tests	A Linter	B Linter	C Fiber	Over-all Avg. for the year
1	3	78.0	72.3	71.2	73.9
2	3	78.5	72.7	71.0	74.1
3	3	77.5	72.1	70.2	73.3
4	3	78.8	73.0	71.8	74.6
5	3	77.7	72.7	71.5	74.0
6	3	78.8	72.3	69.6	73.6
7	3	78.0	72.0	70.0	73.3
9	3	78.1	72.1	70.8	73.7
10	3	78.2	72.3	70.7	73.7
11	3	76.6	72.3	70.7	73.2
Average		78.0	72.4	70.8	73.7

P. D. CRETIEN
W. S. HUDE
R. E. KNIPPLE
T. L. RETTGER
E. H. TENENT
L. N. ROGERS, chairman