Esters of Dimethylethylacetic Acid.—In the course of an investigation being carried on in this Laboratory we had occasion to synthesize several esters of dimethylethylacetic acid. This acid was prepared by treating *tert.*-amylmagnesium chloride with carbon dioxide; esterification was brought about by refluxing the acid and the required alcohol with sulfuric acid as catalyst.

Tert.-amyl chloride was prepared in the following manner. Four thousand cc. of concentrated hydrochloric acid was added, during fifteen to twenty minutes, with mechanical stirring, to 2500 g. of *tert.*-amyl alcohol[1] contained in a 12-liter earthenware crock. The mixture warmed up to 45°; stirring was continued for four hours. The upper layer, *tert.*-amyl chloride containing some unchanged alcohol, was transferred to a 12-liter flask and saturated with dry hydrogen chloride in the presence of anhydrous calcium chloride, the mixture being mechanically stirred throughout the process. Saturation required one and one-half hours; the mixture warmed up to 50°. The calcium chloride was then removed and the *tert.*-amyl chloride mixture was refluxed for an hour. After standing over anhydrous sodium carbonate for twenty-four hours the product was filtered. Twenty cc. of dimethylaniline was then added and the product distilled through a 50-cm. fractionating column. There is continuous evolution of hydrogen chloride during distillation if dimethylaniline is not added. The *tert.*-amyl chloride boiled at 84–86°; the yield was 1960 g., or 65% of the theoretical amount.

Dimethylethylacetic acid was prepared in 60% yield by treating *tert.*-amylmagnesium chloride with carbon dioxide. Our procedure was patterned after that of Puntambeker and Zoellner[2] for the preparation of trimethylacetic acid. The ether from which the dimethylethylacetic acid had been extracted by means of sodium hydroxide solution, by the above directions, contained two by-products—*tert.*-amyl alcohol (b. p. 99–103°, n_D^{23}, 1.4035) and a camphoraceous smelling liquid (b. p. 164–167.5°; n_D^{22}, 1.4335; molecular weight in acetone, 183) which we have not yet investigated.

The esters are colorless liquids. The methyl, ethyl and *n*-propyl esters have menthol–peppermint odors; the *n*-butyl, *n*- and *iso*-amyl esters have but little odor. The refractive indices were determined at 25.0 ± 0.1° by means of an Abbé refractometer; the densities were determined at 25.00 ± 0.02° by means of a pycnometer of about 8-cc. capacity. The weights used in the density calculations were corrected for buoyancy. The boiling points were determined by means of Anschütz thermometers (stem wholly immersed in vapor) which had been standardized by the U. S. Bureau

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[1] Kindly furnished by the Sharples Solvents Corporation.

[2] Puntambeker and Zoellner, "Organic Syntheses," John Wiley and Sons, Inc., New York, **8,** 104 (1928).

of Standards. The molecular weights were calculated using the Lorentz–Lorenz formula. Considerable difficulty was experienced in analyzing these esters, especially the lower ones, low results being obtained repeatedly. Correct results were finally obtained in all cases except that of the methyl ester by using a quartz combustion tube and high temperature.

Table I

Esters of Dimethylethylacetic Acid

Ester	B. p., °C. (746 mm., corr.)	n^{25}_{D}	d^{25}_{4}	Mol. wt.		Analyses			
				Calcd.	Found	Calcd.		Found	
						C, %	H, %	C, %	H, %
Methyl	125 –125.5	1.3991	0.8943	130	134	64.6	10.8	61.0	10.7
								62.8	7.7
Ethyl[a]	141.8–142.2	1.3989	.8601	144	145	66.6	11.2	66.4	11.1
n-Propyl	164 –164.4	1.4040	.8575	158	159	68.4	11.5	68.1	11.4
n-Butyl	184 –184.7	1.4098	.8566	172	173	69.7	11.7	71.0	11.5
n-Amyl	202.5–203.5	1.4140	.8544	186	187	70.9	11.9	71.0	11.8
Iso-Amyl	192.5–196.5	1.4128	.8533	186	187	70.9	11.9	70.6	11.7

[a] Bouveault and Blanc, *Bull. soc. chim.*, [3] **31,** 749 (1904), report boiling point as 141–142° and d^{0}_{4} as 0.883.

Contribution from the
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Reaction of Alizarin and Mercuric Acetate.—The observation here reported was made some six years ago. Since the work is not to be continued, the facts obtained are presented as being of possible interest in anthraquinone chemistry.

Alizarin when heated with excess mercuric acetate gave large amounts of mercurous acetate, indicating an oxidation of the alizarin. The product contained organic mercury. Five runs with varying concentrations and times of heating varying from 30 to 100 hours gave almost identical results. The organic product appeared to be acetoxymercurydihydroxyalizarin. The following average analyses were obtained on the products from the five runs.

Anal. Calcd. for $C_{16}H_{10}O_8Hg$: Hg, 49.8. Found: 49.5, 50.0, 49.8, 49.7, 49.7.

Treatment of the product with hydrochloric acid gave a yellow-red dye with properties similar to those of alizarin.

The mercury compound when treated with the amount of standard sodium hydroxide corresponding to the four hydroxyls gave a bluish-red solution. The solution at 20° then contained 0.2 g. per 100 cc. Warming the solution changed the color to red and precipitated some material which dissolved again on cooling. Both the cold and hot solutions were neutral to litmus.