

phenylacetic acid was isolated, m. p. 262–264°. On esterification with diazomethane, 12 mg. of methyl triphenylacetate was obtained, m. p. 185–187°.

The neutral fraction, recrystallized from benzene-petroleum ether, yielded 25 mg. (0.083 millimole) of 1,1,1-triphenylbutanone-3, m. p. 141–142°, no depression with an authentic sample prepared according to Conant and Scherp.¹

No other products were obtained from the addition product, which therefore appears to be 4-methyl-1,1,1,6,6,6-hexaphenyl-hexadiene-2,3.

Study of the reaction is being extended to various substituted enynes and dienyne, and to conjugated dienes with substituents at the terminal carbon atoms of the conjugated system. As is well known⁶ the presence of terminal substituents limits the use of maleic anhydride for characterizing butadienes. It is hoped that triphenylmethyl, which seems not to be subject to this limitation,¹ will be useful for this purpose and for the characterization of enynes and dienyne, since the reaction in this case proceeded smoothly, and the product was readily isolated.

(6) (a) Bacon and Farmer, *J. Chem. Soc.*, 1065 (1937); (b) Bachman and Goebel, *THIS JOURNAL*, **64**, 787 (1942).

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NEW COMPOUNDS

Diethyl Acetal of 3-Methylbuten-3-ol-1

The method used was the orthoformate synthesis, independently discovered by Tschitschibabin and Bodroux.¹ To an ethereal solution of the Grignard reagent prepared from 18.1 g. of methyl chloride and 14.4 g. of magnesium, after thirty minutes, 25 g. of ethyl orthoformate was added with stirring and the solution allowed to boil under gentle reflux for six and one-half hours after addition was complete. The bulk of the ether was then distilled and the cooled residue hydrolyzed with ice-water. The acetal was isolated by distillation of the organic layer formed upon

(1) Tschitschibabin, *Ber.*, **37**, 186 (1904); Bodroux, *Compt. rend.*, **138**, 93 (1904).

hydrolysis. The product was a colorless liquid which possessed a sharp odor, b. p. 154–155°; n_D^{20} 1.4098, d_4^{20} 0.8316; yield, 5.9 g. (24%).

The *p*-nitrophenylhydrazone, 2,4-dinitrophenylhydrazone and semicarbazone of the aldehyde were prepared according to the directions of Shriner and Fuson.² The *p*-nitrophenylhydrazone crystallized as golden plates from 95% ethanol, m. p. 157°.

Anal. Calcd. for $C_{11}H_{13}N_3O_2$: N, 19.18. Found: N, 19.08. The 2,4-dinitrophenylhydrazone crystallized as shining red needles from 95% ethanol, m. p. 181°. *Anal.* Calcd. for $C_{11}H_{12}N_4O_4$: N, 21.21. Found: N, 21.14. The semicarbazone crystallized from 95% ethanol as shining white needles, m. p. 204–205°. *Anal.* Calcd. for $C_8H_{11}N_3O$: N, 29.79. Found: N, 30.08.

(2) Shriner and Fuson, "Systematic Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., pp. 145, 148.

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Esters of Methylneopentylacetic Acid

The ethyl, *n*-propyl, *n*-butyl, *n*-hexyl esters of methylneopentylacetic acid¹ were prepared by treating 60-g. (0.34 mole) portions of the acid chloride with an excess (100 g.) of the corresponding alcohols. The acid chloride was prepared from thionyl chloride and the acid. The ester was washed with water and 5% sodium carbonate solution, and dried over freshly dehydrated sodium sulfate. Upon fractionating the ester through a column of 20 theoretical plates, packed with $3/32$ -in. stainless steel helices, constant boiling, constant index material was obtained in each case.

| Ester | Yield, % | n_D^{20} | | Cottrell b. p. at 734 mm. | Mol. refraction | |
|------------------|-------------|------------|----------|---------------------------------|-----------------|--------|
| | | Valentine | d^{20} | | Obs. | Calcd. |
| Ethyl | 89 | 1.4131 | 0.8586 | 176.8 | 49.99 | 50.05 |
| <i>n</i> -Propyl | 75 | 1.4174 | .8569 | 196.6 | 54.65 | 54.67 |
| <i>n</i> -Butyl | 77 | 1.4207 | .8556 | 213.8 | 59.32 | 59.29 |
| <i>n</i> -Hexyl | 87 | 1.4271 | .8545 | 247.2 | 68.59 | 68.53 |

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(1) Whitmore and co-workers, *THIS JOURNAL*, **63**, 2028 (1941).