The precipitate was collected, washed with ethanol, dried and crystallized twice from glacial acetic acid; m.p. 281° dec. This compound is very slightly soluble in ethanol.

Anal. Calcd. for $C_{29}H_{28}N_{10}O_2$: C, 63.69; H, 4.80; N, 25.63. Found: C, 63.19; H, 4.91; N, 25.71.

Chromatographic Separation of *p*-Phenylazophenylsemicarbazone of Trioses.—All the semicarbazones reported here are very soluble in pyridine hence the latter was useful for the preparation of solutions for chromatographic work. A pyridine solution of a mixture of the semicarbazone of glyceraldehyde, dihydroxyacetone and pyruvaldehyde was applied to a strip of Whatman No. 1 filter paper to make a spot 6–8 mm. in diameter. The paper had been washed with ethanol previously. The paper strip was developed with a methanol:water mixture (2:1), descending technique was employed. When the solvent front had traveled 40 cm. or more three distinct spots were observed. The pyruvaldehyde derivative remained at the origin as in the case when it was developed as a single substance. The glyceraldehyde derivative has moved further down than the dihydroxyacetone derivative. When developed individually these two derivatives have the following R_f values (27°): glyceraldehyde derivative, 0.61; dehydroxyacetone, 0.52.

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Thiourea Adduct of 1,2,4,5-Tetramethylbenzene

BY J. W. TETER AND W. P. HETTINGER, JR.

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Although benzene itself and most alkyl benzenes do not form adducts with thiourea, we find that durene readily forms a stable adduct. X-ray diffraction patterns of the solid obtained in adduction experiments show the presence of some thiourea but also clearly indicate adduct formation.

We have used this adduct as a means of isolating durene from fractions of high-octane-reforming products boiling in the neighborhood of 196°, the durene content of which can be estimated by cryoscopic means.

The fact that only durene adduction was observed in the presence of other tetramethylbenzene isomers indicates the selectivity of the reaction. Likewise none of the C_8-, C_9- or C_{11}-methyl substituted benzenes appeared to form adducts approaching the stability of durene.

Experimental

A solution of dry methanol, saturated with thiourea at 25° (150 ml.), was mixed with 26.6 ml. of a solution containing 21.9 volume per cent. durene and 78.1 volume per cent. toluene. Almost immediately a voluminous precipitate of white needles was formed. This solution plus precipitate was then cooled to 3°. The remaining mother liquor contained 91.6% toluene and 8.4% durene on a methanol and thiourea free basis.

The precipitate was collected, dried and weighed (12.9 g.) and analyzed.

Anal. Found: C, 37.2; H, 6.3; N, 26.2; S, 30.3.

X-Ray Examination.—Samples of durene, thiourea and the solid complex were analyzed on a North American Philips X-ray spectrometer employing CuK_α radiation. Table I shows the results of these analyses in terms of d/n.

TABLE I

X-RAY DIFFRACTION PATTERNS

Durene thiourea add.		Durene		Thiourea	
d/n	Int.	d/n	Int.	d/n	Int.
7.61	10	....	..	....	..
....	..	6.44	100	....	..
5.80	20	....	..	....	..
....	..	5.29	14	....	..
....	..	5.20	24	....	..
....	..	5.02	10	....	..
4.49	15	....	..	4.60	10
4.438	100	....	..	4.435	100
....	..	4.38	5	....	..
4.308	50	....	..	4.252	100
....	..	3.89	15	....	..
3.85	4	3.85	15	3.82	80
3.64	6	....	..	....	..
3.51	50	....	..	3.50	70
3.37	40	....	..	....	..
....	..	3.22	20	....	..
....	..	....	..	3.13	100
2.93	5	....	..	3.08	70

The data show that a new crystalline substance has been formed. Similarity of the cyclohexane and durene adduct patterns further confirms this.

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4-Methyl-4-(3,5-Dimethylphenyl)-2-Methylpentene-1. An Intermediate Compound in the Synthesis of 1,1,3,3,4,6-Hexamethylindan

BY PHILIPPE TEYSSIÉ AND GEORGES SMETS

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During the course of an investigation on intramolecular cyclization in polymeric systems,[1] the synthesis of several reference substances has been carried out in order to allow an infrared spectrometric determination of the structure of this new class of polymers. For this purpose, 1,1,3,3,4,6-hexamethylindan has been prepared according to the method of Smith and Spillane[2]: reaction of methyl (3,5-dimethylphenyl)-isovalerate (I) with methylmagnesium iodide, and subsequent dehydration of the crude carbinol II in the presence of sulfuric acid. Owing to its insensitivity to ozone and alkaline permanganate in the cold, a structure of 1,1,3,3,4,6-hexamethylindan (IV) was assigned[2] to the hydrocarbon obtained. However, the same procedure in our laboratory gave a hydrocarbon different from that described by Smith and Spillane; it has been proved to be 4-methyl-4-(3,5-dimethylphenyl)-2-methylpentene-1 (III). Moreover, in the presence of aluminum chloride, internal cyclization occurs and the pentene III is trans-

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[1] Ph. Teyssié and G. Smets, Friedel–Crafts Reactions on Polyvinyl Chloride, *J. Polymer Sci.*, in press.

[2] L. I. Smith and J. L. Spillane, THIS JOURNAL, **65**, 202 (1943).