NEW COMPOUNDS

TABLE I SUBSTITUTED BIS-(ADVI OVV)-METHANDS (P)-CH-

	R	Vield, %	n ²⁵ D	M. p., °C.	°C. ^{B.}	p. Mm.	Formula	Carbo Caled.	n, % Found	Hydro Calcd,	gen, % Found	Chlori Calcd.	ne, % Found
1	o-Allylphenoxy	67.1	1.5580		180181	1	C19H202	81.49	81.63	7,14	7.14		
2	2-Allyl-4-chlorophenoxy	29.6	1.5769		202 - 203	2.8	C19H18Cl2O2					20.3	20.5
3	2-Allyl-6-chlorophenoxy	52.6	1.5695		200 - 202	4.1	C19H18Cl2O2					20.30	20.29
4	2-Allyl-6-cyclohexylphenoxy	25.6	1.5567		241 - 242	2.7	C81H40O2	83.70	83.40	9.00	9.02		
5	2-Allyl-4,6-dichlorophenoxy	63.8		51-52	212 - 215	2.6	C18H16Cl4O2					33.9	33.43
6	2-Allyl-4-methoxyphenoxy	31	1.5612		193-195	1.3	C21H24O4	74.09	74.09	7.11	7.20		
7	2-Allyl-p-toloxy	88,8	1.5528		220-224	5.9	$C_{21}H_{24}O_2$	81.8	82.25	7.85	8.27		
8	p-n-Butoxyphenoxy	58.7		68-70	189-191	1.3	C21H28O4	73.2	73.42	8.2	8.44		
9	2-s-Butyl-4-chlorophenoxy	72.5	1.5477		199-201	2.7	C21H25Cl2O2					18.60	18.60
10	4-s-Butyl-2-chlorophenoxy	78.1	1.5471		218-219	3.6	C21H26Cl2O2					18.60	18.47
11	o-s-Butylphenoxy ^a	43,3	1.5314		165-166	2.7	C21H28O2	80.72	80 .58	9.03	9.08		
12	4-Chloro-o-toloxy ^{a,d}	46.5		146 - 146.5			C18H14Cl2O2					23.86	23.83
13	6-Chloro-o-toloxy	53.4	1.5664		165 - 168	2.3	$C_{15}H_{14}Cl_2O_2$					23.86	23.37
14	2,6-Dimethoxyphenoxy ^a	37.2		133-135			C17H20O6	63.74	63 .45	6.29	6.28		
15	p-Methoxyphenoxy4,6	55.5		47-50			C15H16O4	69.21	69.15	6.19	6.21		
16	Pentachlorophenoxy ^{b,d}	42.6		261-262			C13H2Cl10O2					65.09	64.96
17	2,3,4,6-Tetrachlorophenoxy ^{c,d}	29.8		160.5-161.5			C18H4Cl8O2					59.61	59. 84
18	2,4,5-Trichlorophenoxy ⁴	51.3		124-126			C13H6Cl6O2					52.04	52.17

^a Recrystallized from ethanol. ^b Recrystallized from ehlorobenzene. ^c Recrystallized from 60-100° petroleum ether. ^d These compounds were not distilled.

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layer was distilled to remove the chlorobenzene and finally the product, 101 g., was distilled at 212-215° (2.6 mm.), m. p. 51-52°, 63.8% yield.

Anal. Calcd. for $C_{19}H_{16}Cl_4O_2$: Cl, 33.9. Found: Cl, 33.43.

(4) Wilson Baker, J. Chem. Soc., 1765 (1931).

ORGANIC RESEARCH LABORATORY THE DOW CHEMICAL CO. MIDLAND, MICH.

RECEIVED JUNE 10, 1950

Tris-(*m*-trifluoromethylphenyl)-fluorosilane and Tetrakis-(*m*-trifluoromethylphenyl)-silane

Silicone tetrafluoride, generated by the addition of 100 ml. of concd. sulfuric acid to a mixture of 30 g. of sodium silicofluoride and 10 g. of silicon dioxide, was bubbled slowly into a solution of *m*-trifluoromethylphenylmagnesium bromide prepared from 112.0 g. (0.5 mole) of *m*-bromobenzotrifluoride¹ and 12.1 g. of magnesium in 250 ml. of anhydrous ether. The reaction mixture was allowed to stand at room temperature and with stirring for two days. The reaction mixture was hydrolyzed and additional quantities of ether were used to extract the desired products from the partially gelatinous aqueous phase. The ethereal extract after drying was distilled and 1-foot column.

Tris-(*m*-trifluoromethylphenyl)-fluorosilane was isolated as the fraction boiling at $168-174^{\circ}$ (1-2 mm.), $n^{25}D$ 1.4859, d^{25} 1.4008; *MR*p calcd. 97.68, found 98.84. The yield was 18.8 g. or 23.4%.

Anal.² Calcd. for $C_{21}H_{12}F_{16}Si$: C, 52.28; H, 2.50. Found: C, 52.88; H, 2.48.

From the high boiling fractions of several preparations of tris-(m-trifluoromethylphenyl)-fluorosilane there was obtained on fractionation the tetraarylsilane, b. p. 197–200° (2–3 mm.). Several crystallizations from methanol

gave the pure tetrak is-(*m*-trifluoromethylphenyl)-silane melting at $102-103^{\circ}$.

Anal,² Caled. for $C_{28}H_{16}F_{12}Si$: C, 55.26; H, 2.65; mol. wt., 608. Found: C, 55.22; H, 2.57; mol. wt. (Rast method), 571, 572.

DEPARTMENT OF CHEMISTRY DUQUESNE UNIVERSITY

PITTSBURGH, PENNA.

ERSITY H. HARRY SZMANT NNA. JOSEPH F. ANZENBERGER RECEIVED JUNE 1, 1950

4-Benzyloxyacetophenone and -phenylmethylcarbinol

A vigorously agitated mixture of 59 g. (0.43 mole) of 4-hydroxyacetophenone,¹ 56.9 g. (0.45 mole) of benzyl chloride, 150 g. of anhydrous potassium carbonate and 350 cc. of methyl ethyl ketone was refuxed for 12 hours. Water (750 cc.) was added and the product extracted with ether. The solution was washed with alkali and water, dried and concentrated. The oily residue crystallized spontaneously. After trituration with petroleum ether, the 4-benzyloxyacetophenone was recrystallized from isopropyl alcohol. It melted at 96-97°; yield 63 g. (64.3%).

Anal. Caled. for $C_{1b}H_{14}O_2$: C, 80.6; H, 6.2. Found: C, 80.3; H, 6.1.

In an efficient column, 9.3 g. of 4-benzyloxyacetophenone was reduced with 8.5 g. of aluminum isopropoxide and 150 cc. of isopropyl alcohol in the usual manner, replacing the isopropyl alcohol from time to time. The reaction product was treated with an aqueous solution of 15 g. of potassium hydroxide and the precipitate extracted with ether. 4-Benzyloxyphenylmethylearbinol boiled at 195-200° at 16 mm. and solidified spontaneously. It was recrystallized from a mixture (1:1) of benzene and petroleum ether and melted at 79-80°; yield, 3.5 g. (37.6%).

Anal. Calcd. for $C_{16}H_{16}O_2$: C, 78.9; H, 7.0. Found: C, 78.9; H, 7.0.

GROSVENOR LABORATORY LONDON, S. W. 1, ENGLAND RECEIVED JULY 5, 1950

(1) Noller and Adams, THIS JOURNAL, 46, 1892 (1924).

⁽¹⁾ We acknowledge with thanks the generous gift of benzotrifluoride from the Hooker Chemical Co.

⁽²⁾ Microanalyses by the Micro-Analytical Laboratory, University of Pittsburgh.