TABLE I

SUBSTITUTED BIS-(ARYLOXY)-METHANES, $(R)_2CH_2$

	R	Yield, %	$n^{25}D$	M. p., °C.	B. p. °C.	B. p. Mm.	Formula	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Chlorine, % Calcd.	Chlorine, % Found
1	*o*-Allylphenoxy	67.1	1.5580		180–181	1	$C_{19}H_{20}O_2$	81.49	81.63	7.14	7.14		
2	2-Allyl-4-chlorophenoxy	29.6	1.5769		202–203	2.8	$C_{19}H_{18}Cl_2O_2$					20.3	20.5
3	2-Allyl-6-chlorophenoxy	52.6	1.5695		200–202	4.1	$C_{19}H_{18}Cl_2O_2$					20.30	20.29
4	2-Allyl-6-cyclohexylphenoxy	25.6	1.5567		241–242	2.7	$C_{31}H_{40}O_2$	83.70	83.40	9.00	9.02		
5	2-Allyl-4,6-dichlorophenoxy	63.8		51–52	212–215	2.6	$C_{19}H_{16}Cl_4O_2$					33.9	33.43
6	2-Allyl-4-methoxyphenoxy	31	1.5612		193–195	1.3	$C_{21}H_{24}O_4$	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	$C_{21}H_{28}O_4$	73.2	73.42	8.2	8.44		
9	2-*s*-Butyl-4-chlorophenoxy	72.5	1.5477		199–201	2.7	$C_{21}H_{26}Cl_2O_2$					18.60	18.60
10	4-*s*-Butyl-2-chlorophenoxy	78.1	1.5471		218–219	3.6	$C_{21}H_{26}Cl_2O_2$					18.60	18.47
11	*o*-*s*-Butylphenoxy[a]	43.3	1.5314		165–166	2.7	$C_{21}H_{28}O_2$	80.72	80.58	9.03	9.08		
12	4-Chloro-*o*-toloxy[a,d]	46.5		146–146.5			$C_{15}H_{14}Cl_2O_2$					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			$C_{17}H_{20}O_6$	63.74	63.45	6.29	6.28		
15	*p*-Methoxyphenoxy[4,a]	55.5		47–50			$C_{15}H_{16}O_4$	69.21	69.15	6.19	6.21		
16	Pentachlorophenoxy[b,d]	42.6		261–262			$C_{13}H_2Cl_{10}O_2$					65.09	64.96
17	2,3,4,6-Tetrachlorophenoxy[c,d]	29.8		160.5–161.5			$C_{13}H_4Cl_8O_2$					59.61	59.84
18	2,4,5-Trichlorophenoxy[a]	51.3		124–126			$C_{13}H_6Cl_6O_2$					52.04	52.17

[a] Recrystallized from ethanol. [b] Recrystallized from chlorobenzene. [c] Recrystallized from 60–100° petroleum ether. [d] These compounds were not distilled.

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.

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

ORGANIC RESEARCH LABORATORY
THE DOW CHEMICAL CO. EZRA MONROE
MIDLAND, MICH. CLARE R. HAND

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*-bromobenzotrifluoride[1] 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 after removal of the ether the residue was fractionated in a 1-foot column.

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

Anal.[2] Calcd. for $C_{21}H_{12}F_{10}Si$: 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 tetrakis-(*m*-trifluoromethylphenyl)-silane melting at 102–103°.

Anal.[2] Calcd. 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 H. HARRY SZMANT
PITTSBURGH, PENNA. JOSEPH F. ANZENBERGER

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[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.

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 refluxed 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. Calcd. for $C_{15}H_{14}O_2$: 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-Benzyloxyphenylmethylcarbinol** 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 $C_{15}H_{16}O_2$: C, 78.9; H, 7.0. Found: C, 78.9; H, 7.0.

GROSVENOR LABORATORY
LONDON, S. W. 1, ENGLAND M. SULZBACHER

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