*Anal.* Calcd. for $C_{12}H_{17}Br$: C, 59.74; H, 7.06; Found: C, 59.56; H, 6.84.

**3,5-Di-*i*-propylphenyltrimethylsilane.**—The bromide was coupled with trimethylchlorosilane by sodium according to the procedure employed for the diethyl compound. The product, which was fractionated through a Todd column, boiled at 94–95° (3 mm.), $n^{20}\text{D}$ 1.4894, $d^{20}_4$ 1.8605; *MR* found 78.60, *MR* calcd. 77.90.

*Anal.* Calcd. for $C_{15}H_{26}Si$: C, 76.84; H, 11.18. Found: C, 76.54; H, 11.00

**3,5-Di-*t*-butyltoluene**[42] was prepared in 46% yield. The product, which was fractionated through a Todd column, boiled at 94.5–95° (5 mm.) (reported[42] b.p. 98° (6 mm.).

**3,5-Di-*t*-butylbenzoic Acid.**[43]—Oxidation of the di-*t*-butyltoluene gave the desired acid in 64% yield, m.p. 170–171.5° (lit.[43] m.p. 172°).

**3,5-Di-*t*-butylaniline.**[44]—A Schmidt reaction on the acid gave the amine (m.p. 52.5–54°) in 88% yield (lit.[44] m. p. 50.5–53°).

**3,5-Di-*t*-butyliodobenzene** was prepared in 33% yield according to the procedure described in the literature.[44] The iodide melted at 66–67° (lit.[44] m.p. 67–68°).

**3,5-Di-*t*-butylphenyltrimethylsilane.**—A solution of 25.25 g. (0.08 mole) of 3,5-di-*t*-butyliodobenzene in 75 ml. of ether was added at 0–10° to a solution of *n*-butyllithium prepared from 19.2 g. (0.14 mole) of *n*-butyl bromide and 2.00 g. (0.28 g. atom) of lithium wire. Stirring was continued at 0° for 15 minutes. The cooling was removed and 15.25 g. (0.14 mole) of trimethylchlorosilane in 25 ml. of ether was added dropwise. The mixture was stirred at room temperature for three hours then refluxed for four hours. Hydrolysis was effected by pouring onto crushed ice which was mixed with several grams of sodium bicarbonate. The ethereal layer was washed successively with several portions of water, aqueous sodium thiosulfate and finally water. The solution was dried over Drierite. Upon distillation, iodine distilled over with the product. The product was dissolved in 35–37° petroleum ether, washed with thiosulfate, dried, and distilled. This procedure was repeated three times before a product was obtained free from iodine. Final purification was achieved by distillation through a Todd cloumn. The product boiled at 82–83°at 1 mm., $n^{20}\text{D}$ 1.4854, $d^{20}_4$ 0.8573; *MR* found 87.75, *MR* calcd. 87.15.

*Anal.* Calcd. for $C_{17}H_{30}Si$: C, 77.80; H, 11.52. Found: C, 78.04; H, 11.62.

**General Kinetic Procedure.**—The purification of materials, thermostat, dilatometers and procedure were described in a previous paper from this Laboratory.[9]

**Acknowledgment.**—The authors are grateful to the National Science Foundation and the Ethyl Corporation, whose financial assistance made this work possible.

LAFAYETTE, IND.

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[42] J. Greuze, C. Ruinard, J. Soeterbroek, P. E. Verkade and B. M. Wepster, *Rec. trav. chim.*, **75**, 301 (1956).

[43] W. VanHartingsveldt, P. E. Verkade and B. M. Wepster, *ibid.* **75**, 349 (1956).

[44] J. Burgers, W. Van Hartingsveldt, J. Kuelen, P. Verkade, H. Visser and B. M. Wepster, *ibid.*, **75**, 1327 (1956).

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[CONTRIBUTION FROM THE CHEMICAL LABORATORIES OF PURDUE UNIVERSITY]

# A New Method for Determining the Reactivity of a Particular Ring Position in an Aromatic System

BY ROBERT A. BENKESER, DONALD I. HOKE AND RICHARD A. HICKNER

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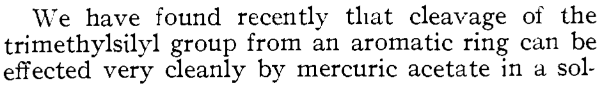
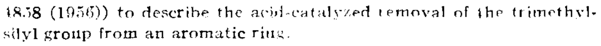
It has been found that the trimethylsilyl group can be removed cleanly from an aromatic ring by mercuric acetate in glacial acetic acid (mercuridesilylation). The mercuriacetate enters the ring at the position originally occupied by the silicon grouping. The rates of mercuric acetate cleavage of the isomeric tolyl- and xylyltrimethylsilanes are reported. The relative order of the ease of cleavage of these compounds parallels closely that which was observed in protodesilylations, and can be rationalized in terms of a combined steric and electronic effect. A plot of log $P_t/m_t$ *vs.* log $p_t$ for mercuridesilylation obeys the linear "selectivity" relationship demonstrated by H. C. Brown and co-workers.

In previous publications from this Laboratory the protodesilylation[1] of various aryltrimethylsilanes was studied.[2] This reaction has been shown to resemble aromatic electrophilic substitution very closely and has proved to be a valuable tool in determining the electrical effect at a particular ring position in an aromatic nucleus.



(protodesilylation)

We have found recently that cleavage of the trimethylsilyl group from an aromatic ring can be effected very cleanly by mercuric acetate in a solvent of glacial acetic acid. In such cases the trimethylsilyl group is replaced by a mercuriacetate group rather than by a proton. It was of interest to determine whether the results obtained in protodesilylations[2] could be correlated in any manner with the mercuridesilylation results. To test this point the isomeric tolyl- and xylyltrimethylsilanes were chosen for study since protodesilylations had been carried out on the members of both of these series previously. Table I lists all the compounds included in the present study along with their physical constants.



(mercuridesilylation)

Table II lists the rate constants and partial relative rates (P.R.R.) for the tolyl series. These cleavages were carried out as pseudo first-order in silane, the mercuric acetate being present in tenfold excess.

It was not possible to carry out cleavages in the xylyl series under the same conditions since the rates proved to be too rapid to follow. Consequently the latter were run pseudo first-order in mercuric acetate. The rate data for the xylyl series are listed in Table III.

It will be noted that the mercuridesilylation reaction provides a new synthetic route to aromatic

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[1] This term was introduced by Prof. C. Eaborn (*J. Chem. Soc.*, 1858 (1956)) to describe the acid-catalyzed removal of the trimethylsilyl group from an aromatic ring.

[2] (a) R. A. Benkeser and H. R. Krysiak, THIS JOURNAL, **76**, 6353 (1954); (b) R. A. Benkeser, R. A. Hickner and D. I. Hoke, *ibid.*, **80**, 2279 (1958); (c) R. A. Benkeser, O. H. Thomas and W. A. Schroeder, *ibid.*, **80**, 2283 (1958); (d) R. A. Benkeser, R. A. Hickner, D. I. Hoke and O. H. Thomas, *ibid.*, **80**, 5289 (1958).

TABLE I

| Product (R = $Me_3Si$-) | $n^{20}_{D}$ Found | $n^{20}_{D}$ Reported | B.p., °C. (mm.) | Yield, % |
|---|---|---|---|---|
| R-Benzene | 1.4907 | 1.4908$^a$ | 167 | 67 |
| *o*-R-Toluene | 1.5034 | 1.5034$^b$ | 196–197 | 67 |
| *m*-R-Toluene | 1.4922 | 1.4922$^b$ | 189 | 55 |
| *p*-R-Toluene | 1.4915 | 1.4915$^b$ | 192 | 53 |
| 2-R-*p*-Xylene | 1.5050 | 1.5048$^c$ | 215–216 | 55 |
| 2-R-*m*-Xylene | 1.5090 | 1.5090$^c$ | 85–86 (5) | 72 |
| 4-R-*m*-Xylene | 1.5050 | 1.5040$^c$ | 218 | 69 |
| 5-R-*m*-Xylene | 1.4940 | 1.4948$^c$ | 73–74 (4) | 36 |
| 3-R-*o*-Xylene | 1.5110$^d$ | 1.5080$^c$ | 217–218 | 55 |
| 4-R-*o*-Xylene | 1.5007 | 1.5007$^c$ | 113 (30) | 65 |

[a] R. A. Benkeser and P. E. Brumfield, THIS JOURNAL, **73**, 4770 (1951). [b] O. H. Thomas, Ph.D. Thesis, Purdue University, 1955. [c] R. A. Benkeser and H. R. Krysiak, THIS JOURNAL, **76**, 6353 (1954). [d] It will be noted that this refractive index is considerably different from that reported by us previously (ref. *c* above). We have discovered that our earlier sample was contaminated with the 4-trimethylsilyl-*o*-xylene isomer. The amine (Eastman practical grade) was purified as the sulfate (O. H. Emerson and L. I. Smith, THIS JOURNAL, **62**, 141 (1940)). The traces of 4-trimethylsilyl-*o*-xylene could be removed easily with a Todd column by atmospheric distillation.

TABLE II

RATE CONSTANTS AND PARTIAL RELATIVE RATES (P.R.R.) FOR THE CLEAVAGE OF TRIMETHYLPHENYLSILANE AND THE ISOMERIC TRIMETHYLTOLYLSILANES (0.017 *M*) BY MERCURIC ACETATE (0.1788 *M*) IN GLACIAL ACETIC ACID AT 25°

| Compound (R = trimethylsilyl) | Rate constant × $10^3$, min.$^{-1}$ | P.R.R. |
|---|---|---|
| R-Benzene | 1.10 ± 0.07 | 1 |
| *o*-R-Toluene | 11.9 ± .03 | 10.8 |
| *m*-R-Toluene | 2.19 ± .02 | 2.0 |
| *p*-R-Toluene | 12.7 ± .02 | 11.5 |

TABLE III

RATE CONSTANTS AND PARTIAL RELATIVE RATES FOR THE CLEAVAGE OF TRIMETHYLPHENYLSILANE AND THE ISOMERIC TRIMETHYLTOLYL- AND TRIMETHYLXYLYLSILANES (0.4000 *M*) BY MERCURIC ACETATE (0.04*M*) IN GLACIAL ACETIC ACID AT 25°

| Compound (R = trimethylsilyl) | Rate constant × $10^3$, min.$^{-1}$ | P.R.R. |
|---|---|---|
| R-Benzene | 0.616 ± 0.002 | 1.0 |
| *o*-R-Toluene | 6.95 ± .01 | 11.3 |
| *m*-R-Toluene | 1.60 ± .02 | 2.59 |
| *p*-R-Toluene | 6.60 ± .02 | 10.7 |
| 3-R-*o*-Xylene | 25.5 ± .4 | 43.0 |
| 4-R-*o*-Xylene | 16.8 ± .1 | 27.2 |
| 4-R-*m*-Xylene | (99)$^a$ | (160)$^a$ |
| 2-R-*m*-Xylene | Too fast to measure | |
| 5-R-*m*-Xylene | 2.19 ± 0.08 | 3.55 |
| 2-R-*p*-Xylene | 15.0 ± .6 | 24.3 |

[a] Determined by isolating the cleavage product.

mercurials. The mercuriacetate group is introduced unequivocally into the position originally occupied by the trimethylsilyl group. In Table IV are listed the melting points and analyses of the xylylmercurials which were prepared by this method.

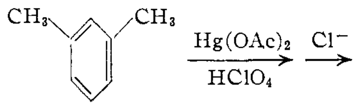
TABLE IV

MELTING POINTS AND ANALYSES OF THE ISOMERIC XYLYLMERCURIC CHLORIDES

| Compound (R = HgCl) | M.p., °C. (uncor.) | Analyses,$^a$ % Carbon | Analyses,$^a$ % Hydrogen |
|---|---|---|---|
| 3-R-*o*-Xylene | 160.5–161.0 | 27.96 | 3.05 |
| 4-R-*o*-Xylene | 194.5–195.5 | 28.22 | 2.95 |
| 2-R-*p*-Xylene$^b$ | 182.5–183.0 | 28.09 | 2.90 |
| 2-R-*m*-Xylene | 157–158 | 28.02 | 2.77 |
| 4-R-*m*-Xylene$^c$ | 159.5–160.0 | 27.72 | 2.85 |
| 5-R-*m*-Xylene | 198.0–198.5 | 27.72 | 2.99 |

[a] Calculated for $C_8H_9HgCl$: C, 28.16; H, 2.66. [b] R. E. McClure and A. Lowry, THIS JOURNAL, **53**, 319 (1931), report a melting point of 183° for this compound. [c] Mixed melting point with a sample obtained by mercurating *m*-xylene gave no depression.

**Results and Discussion.**—In order to be certain that the expansion in volume noted in the dilatometers[3] was an accurate measure of the rate cleavage of the trimethylsilyl group, the aromatic mercurial produced was isolated as the mercurichloride in each instance. In every case where the organomercurial was a known compound, the melting points agreed well with the recorded literature value. In the case of the xylyl mercurials, all but one were new compounds (see Table IV). One of these (4-chloromercuri-*m*-xylene) we synthesized independently by mercurating *m*-xylene in a mixture of 70% perchloric acid and acetic acid.



The product was identical in mixed melting point with the material obtained from the cleavage of 4-trimethylsilyl-*m*-xylene.[4]

Difficulties were encountered in following the rates of cleavage of 2-trimethylsilyl-*m*-xylene and 4-trimethylsilyl-*m*-xylene. In the former case a rapid increase in volume was expected in the dilatometer. Instead only a slow rise was noted over an extended period of time. Also the increase in volume was considerably larger than that observed in the case of the other silanes. When a cleavage of this compound was attempted on a large scale using identical conditions (time, temperature, concentrations, etc.) to those employed in the kinetic runs, an 88% yield of organomercurial was obtained in five minutes. It was obvious that this reaction was so rapid that it was essentially over by the time the dilatometers were filled. The slow rise which had been noted may well have been caused by the gradual solvolysis of the silane (present in excess) by the glacial acetic acid. Thus when a sample of the silane was refluxed with glacial acetic acid some cleavage occurred as was evidenced by the isolation of *m*-xylene.

Similarly the cleavage of 4-trimethylsilyl-*m*-xylene was too rapid to measure by the dilatometric procedure. When a 7-gram sample of this isomer was treated with mercuric acetate in glacial acetic acid with concentrations identical to those employed in the kinetic runs, a 51% yield of organomercurial was obtained in seven minutes. It was obvious that the cleavage was well over half completed by the time the dilatometers were filled. Accordingly, an approximate half-life of seven minutes was assumed in this case and the rate constant calculated on this basis (see Table III).

It will be noted from Table III that in the xylyl

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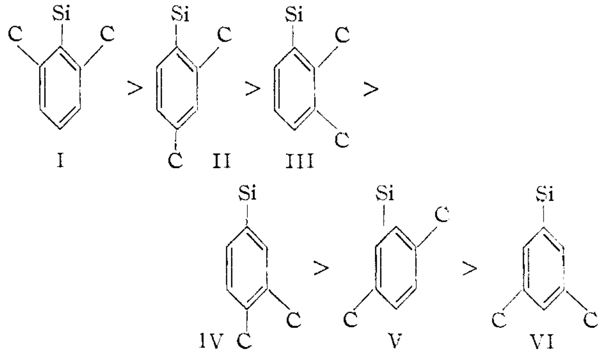
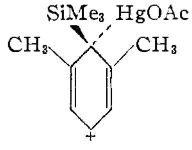
(3) See ref. 2a for a description of the dilatometric method used to follow these rates.

(4) The cleavage products were always converted to chlorides for ease of handling (see Experimental).

series the rates of mercuridesilylation decrease in the order



This is essentially the same order which was observed in the acid cleavages.[2a] It is readily apparent that steric acceleration is again playing an important role in these mercury cleavages. As noted above the 2-trimethylsilyl-*m*-xylene (I) cleaved so rapidly its rate could not be measured. This rapid rate of cleavage obviously is caused by a relief of strain in the transition state wherein the bulky trimethylsilyl group is thrown out of the plane of the two adjacent methyl groups. It



should be kept in mind that the mercuriacetate group probably is linear and hence the final organomercurial will also be less strained than the starting silane. The rapid cleavage of 4-trimethylsilyl-*m*-xylene (II) is best explained in terms of an electronic effect. The trimethylsilyl group in this compound is situated *ortho* and *para* to two methyl groups and hence is extremely vulnerable to displacement by an electrophilic group. Of extreme interest is the relative positions of 3-trimethylsilyl-*o*-xylene (III) and 2-trimethylsilyl-*p*-xylene (V). Each of these compounds has a methyl group *ortho* and one *meta* to the trimethylsilyl. However, the steric acceleration afforded by the buttressing effect of a 1-2-3 arrangements of groups in III causes this compound to cleave faster. It is not surprising that 3-trimethylsilyl-*m*-xylene (VI) cleaves the least rapidly of any compound in the series. In this case only the inductive effect of the two *m*-methyl groups assists the reaction.

In Fig. 1 the partial relative rates[5] for the mercuric acetate cleavage are plotted against the partial relative rates for acid cleavage.[2a,6] The correlation is excellent except in those cases where the rate is seriously affected by steric acceleration.

In 1953, Brown and Nelson[7] introduced the concept of variation in "activity" of the attacking groups in electrophilic substitutions. A linear relationship was demonstrated between log $p_f/m_f$ and log $p_f$[8] for several electrophilic substitutions. While the original data for mercuration did not fall accurately on this line[7] this was later remedied.[9] It is interesting that by using values derived from Table II a point for mercuridesilylation can be plotted which falls very close to this line. In Fig. 2 a portion of the graph is reproduced showing the points for protodesilylation and mercuridesilylation along with the points determined by Brown and McGary[9] for uncatalyzed mercuration. The agreement between their values and ours is excellent.

## Experimental

**Preparation of Compounds.**—The trimethylarylsilanes were prepared by the reaction of the appropriate aryllithium compound and trimethylchlorosilane. The physical constants and yields of these silanes are summarized in Table I.

**Purification of Materials.**—Glacial acetic acid was distilled from potassium permanganate and the calculated amount of acetic anhydride. The mercuric acetate (Baker and Adamson reagent grade) was used without further purification.

**Thermostat and Dilatometers.**—The thermostat and dilatometers used in this study have been described in a previous paper from this Laboratory.[2a]

**General Kinetic Procedure.**—For the rate determinations which were pseudo first-order with respect to silane, 2.849 g. of mercuric acetate was weighed out on an analytical balance and dissolved in about 45 ml. of glacial acetic acid. The silane (0.12–0.15 g.) was weighed into a 50-ml. volumetric flask and the latter was then suspended in the constant temperature bath. At zero time the mercuric acetate solution was poured into the volumetric flask, the glacial acetic acid rinsings added, and the flask filled to the mark with glacial acetic acid. The flask then was removed from the bath, stoppered, and shaken vigorously.

For the rate determinations which were pseudo first-order in mercuric acetate a 0.1 *M* solution of mercuric acetate in glacial acetic acid was prepared. A quantity of silane was weighed into a 50-ml. volumetric flask on an analytical balance so that the silane concentration would be 0.400 *M*. The flask then was suspended in the constant temperature bath. At zero time a 20-ml. aliquot of the mercuric acetate solution was added to the silane and the flask filled to the mark with glacial acetic acid. The flask now was removed from the bath, stoppered and shaken vigorously. The solutions were transferred rapidly by suction to the dilatometer. Readings of the meniscus height were recorded with the aid of a cathetometer which could be read to 0.01 cm. All runs exhibited an expansion in volume upon cleavage, the increase amounting to 0.3 to 2.3 cm.

Rate constants (pseudo first-order) were determined graphically from a plot of 2 + log ($l_\infty - l_t$) against time, $t$, in minutes ($l$ is the meniscus height). The slope of this plot is equal to $-k/2.303$ where $k$ is the pseudo first-order rate constant.

**Rate of Cleavage of 2-Trimethylsilyl-*m*-xylene.**—When the dilatometer was filled with the solution containing this silane (mercuric acetate concentration one-tenth that of the silane) a rapid increase in meniscus height was expected; however, this was not observed. Instead a slow increase, over an extended period of time, was noted. Also the observed increase was considerably larger than expected on the basis of the increase observed with the other trimethylxylylsilanes. The following experiment was carried out to determine whether the cleavage was indeed occurring very rapidly or whether it was slow, as the increase of meniscus height would indicate.

Mercuric acetate (3.6 g., 0.011 mole) was dissolved in 100 ml. of glacial acetic acid and 2 g. (0.011 mole) of 2-trimethylsilyl-*m*-xylene was added. The solution was thoroughly mixed and after five minutes was poured into a solution of 3 g. (0.052 mole) of sodium chloride in 150 ml. of water. The white precipitate of the xylylmercuric chloride was filtered immediately and recrystallized from 95% eth-

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(5) These values are obtained by dividing the rate constant for the cleavage of the compound in question by the rate constant for phenyltrimethylsilane.

(6) The acid-catalyzed cleavage of the tolyltrimethylsilanes is reported in the Ph.D. thesis of O. H. Thomas, Purdue University, 1955.

(7) H. C. Brown and K. L. Nelson, This Journal, 75, 6292 (1953).

(8) $p_f$ refers to the partial rate factor for the *p*-position of toluene; $m_f$ refers to the partial rate factor for the *m*-position of toluene.

(9) H. C. Brown and C. W. McGary, Jr., This Journal, 77, 2300 2306 (1955).

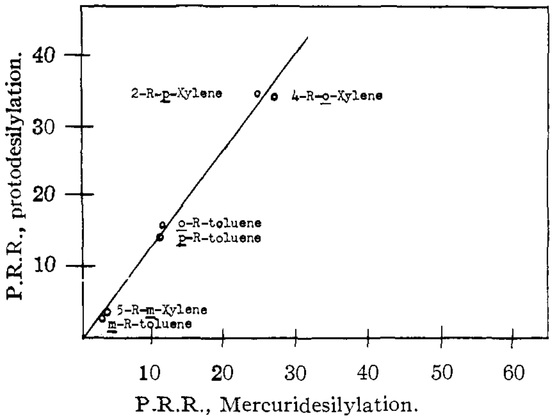

Fig. 1.—Correlation of mercuridesilylation data with protodesilylation data.

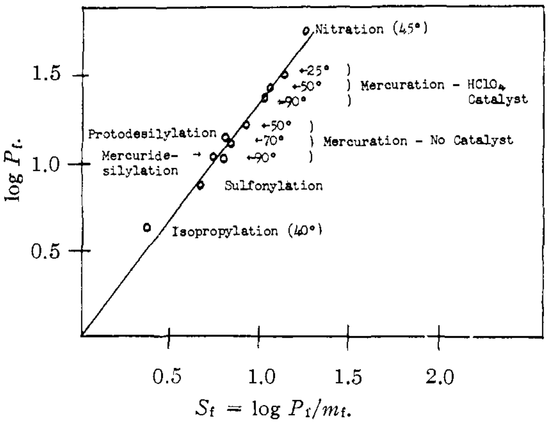

Fig. 2.—Correlation of mercuridesilylation data with the substitution data for toluene.

anol. There was obtained 3.4 g. (88%) of a white crystalline solid melting at 154–155°. This indicated that the cleavage reaction was taking place very rapidly and that a slow side reaction, occurring subsequent to the desilylation, was responsible for the slow increase in volume noted in the dilatometer.

**Solvolysis of 2-Trimethylsilyl-*m*-xylene.**—A solution of 5 g. (0.028 mole) of 2-trimethylsilyl-*m*-xylene in 150 ml. of glacial acetic acid was refluxed for 48 hours and then allowed to stand at room temperature an additional 45 hours. The solution next was poured into one liter of water and extracted with six 50-ml. portions of ether. The combined ether extracts were washed with 100 ml. of cold water, dried with Drierite and distilled. A two component mixture distilled at 90–100°. One component was insoluble in water. This was separated, washed with sodium bicarbonate solution, water, and finally dried with Drierite; yield 3 g., $n^{20}D$ 1.4468.

Following the procedure of Shriner and Fuson[10] for the nitration of hydrocarbons, 1 ml. of the above liquid was nitrated. The solid product was recrystallized from absolute ethanol. The ethanol solution was not cooled below room temperature to avoid obtaining a mixture of the di- and trinitro compounds. The material isolated in this manner melted at 180–181°. A sample of authentic *m*-xylene was nitrated and recrystallized in the same manner (m.p. 180–181°). A mixed melting point of the two substances gave no depression. The reported[11] melting point for 2,4,6-trinitro-*m*-xylene is 182°.

**2,4-Dimethylphenylmercuric acetate.**—Mercuric acetate (32 g., 0.1 mole) and 3 ml. of 70% perchloric acid were added, with stirring, to a solution of 15 g. (0.15 mole) of *m*-xylene in 200 ml. of glacial acetic acid. The solution was stirred until a precipitate started to form. It then was poured into twice its own volume of water and the white solid filtered. The latter was recrystallized from aqueous ethanol and melted at 129.8–130.8° (reported[12] 126–127°).

**2,4-Dimethylphenylmercuric Chloride.**—Sodium chloride was added to the filtrate from the recrystallization of the 2,4-dimethylphenylmercuric acetate. The white solid was filtered and recrystallized from 95% ethanol (m.p. 155–156°). The analysis is given in Table IV.

**Isolation of Cleavage Products.**—A solution of 15 g. (0.1 mole) of trimethylphenylsilane and 3.6 g. (0.011 mole) of mercuric acetate in 200 ml. of glacial acetic acid was stored at room temperature for the length of time required for completion of the cleavage. At the end of this time the solution was poured into excess sodium chloride solution. The white precipitate was filtered and dried. The yield of phenylmercuric chloride was 3.25 g. (92%), m.p. 249.5–250.5° (reported[13] 251°).

Following the above procedure *p*-tolylmercuric chloride, m.p. 235–236° (reported[14] 236–237°) and *o*-tolylmercuric chloride, m.p. 141° (reported[14] 143°) were prepared by cleavage of trimethyl-*p*-tolylsilane and trimethyl-*o*-tolylsilane, respectively.

A similar procedure was used to prepared the unknown xylylmercuric chlorides. Their physical constants and analysis are given in Table IV.

**Rate of Cleavage of 4-Trimethylsilyl-*m*-xylene.**—A solution of 7.12 g. (0.04 mole) of 4-trimethylsilyl-*m*-xylene and 1.28 g. (0.004 mole) of mercuric acetate in 100 ml. of glacial acetic acid was poured into an equal volume of water after standing 7 minutes. Excess sodium chloride was added, and the white precipitate of 2,4-dimethylphenylmercuric chloride was filtered immediately and dried. There was obtained 0.68 g. (51%). This indicates a half-life of about 7 minutes and not 21 minutes derived from the rate data. Thus the rate is too fast to be measured accurately by the dilatometric procedure. Using a value for the 7 minutes a rate constant of $99 \times 10^{-3}$ min.$^{-1}$ is obtained.

**Acknowledgment.**—The authors are deeply grateful to the National Science Foundation whose financial assistance made this work possible.

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[10] R. L. Shriner and R. C. Fuson, "The Systematic Identification of Organic Compounds," 3rd ed., John Wiley and Sons, Inc., New York, N. Y., 1948, p. 197.

[11] L. F. Fieser, R. C. Clapp and W. H. Daudt, THIS JOURNAL, **64**, 2052 (1942).

[12] K. Hiratuka, *J. Chem. Soc. Japan*, **58**, 1051 (1937); *C. A.*, **33**, 157 (1939).

[13] A. N. Nesmeyanov and O. A. Reutov, *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 316 (1948).

[14] A. N. Nesmeyanov, *Ber.*, **62**, 1010 (1929).