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HALOMETHYL–METAL COMPOUNDS
VIII[a]. THE REACTION OF PHENYL(TRIHALOMETHYL)MERCURY COMPOUNDS WITH HYDROGEN CHLORIDE[b]

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In our study[2] of the reaction of phenyl(bromodichloromethyl)mercury with n-butyl alcohol in benzene at 80°, two products were formed (n-butyl chloride and n-butyl formate) which could be rationalized as deriving from initially formed n-$C_4H_9OCCl_2H$ (eqn. 1 and 2). Two other volatile products, benzene and chloroform,

$$C_6H_5HgCCl_2Br + C_4H_9OH \rightarrow C_4H_9OCCl_2H + C_6H_5HgBr \quad (1)$$

$$C_4H_9OCCl_2H + C_4H_9OH \rightarrow (C_4H_9O)_2CHCl + HCl \quad (2)$$
$$\hookrightarrow C_4H_9Cl + HCOOC_4H_9$$

were present in the reaction mixture. The former most certainly resulted from cleavage of phenyl groups from mercury by hydrogen chloride formed in eqn. (2). Two processes could have led to formation of chloroform: (*i*) Cleavage of CCl_2Br groups from mercury by hydrogen chloride, accompanied or followed by halogen exchange, and (*ii*) insertion of CCl_2 (derived from $C_6H_5HgCCl_2Br$[3]) into the H–Cl bond. It was the purpose of the present investigation to determine the nature of the reaction(s) occurring between phenyl(trihalomethyl)mercury compounds and hydrogen chloride.

A previous study[1] showed that phenyl(bromodichloromethyl)mercury reacts with carboxylic acids as shown in eqn. (3). With weaker acids, such as acetic acid,

$$C_6H_5HgCCl_2Br + RCOOH \xrightarrow{C_6H_6,\ 80^\circ} RCOOCCl_2H + C_6H_5HgBr \quad (3)$$

this reaction proceeded virtually quantitatively as written. With stronger acids, *e.g.*, trichloroacetic acid, the dichloromethyl ester yields were considerably lower, and C_6H_5–Hg cleavage by the acid became a major side reaction. The formation of dichloromethyl esters in reaction (3) was discussed in terms of a CCl_2/RCOOH reaction. Thus a similar competition of cleavage and insertion might be expected with hydrogen chloride. It may also be noted that the formation of CCl_2 and HCl in the pyrolysis of chloroform at 485–600° is thought to be a reversible reaction[4].

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[a] Part VII: ref. 1.
[b] Preliminary communication: ref. 2.
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Furthermore, it has been reported that difluorocarbene reacts with hydrogen chloride in the gas phase to give $HCF_2Cl^{5,6}$.

In the present work it was found that when anhydrous hydrogen chloride was bubbled into a chlorobenzene solution of phenyl(bromodichloromethyl)mercury, benzene and chloroform are indeed produced. The reaction temperature has a very marked effect on the relative amounts of these products which are formed. At 85–88° the benzene/chloroform ratio was approximately 1. This ratio increased with decreasing temperature, and at room temperature the reaction proceeded cleanly as depicted in eqn. (4), with no detectable amounts of chloroform being produced. The results of these experiments are given in Table 1. This very specific C_6H_5–Hg cleavage

$$C_6H_5HgCCl_2Br + HCl \xrightarrow{30°} C_6H_6 + ClHgCCl_2Br \ (95\%) \qquad (4)$$

also could be used to prepare $ClHgCCl_3$ (91%), $ClHgCClBr_2$ (96%) and $ClHgCBr_3$ (96%) by reaction of the appropriate phenyl(trihalomethyl)mercurial with hydrogen chloride at room temperature. Essentially quantitative yields of benzene were realized in these last three reactions, and no HCX_3 was detected.

TABLE 1

REACTION OF $C_6H_5HgCCl_2Br$ (7 MMOLES) AND HCl AT VARIOUS TEMPERATURES IN CHLOROBENZENE

Temp. range (°C)	*C_6H_6 yield (%)*[c]	*$HCCl_3$ yield (%)*	*Reaction time (min)*[a]	*Total wt. of solid (g)*[b]	*M.p. of solid (°C)*
85–88	54	58	12	2.57	
79–81	76	37	10	2.54	
73–75	89	19	20	2.68	157–161 dec.
58–61	85	6	30	2.76	161–163 dec.
42–45	89	0.5	40	2.81	161–163.5 dec.
30–32	93	0	50	2.80	162–164 dec.

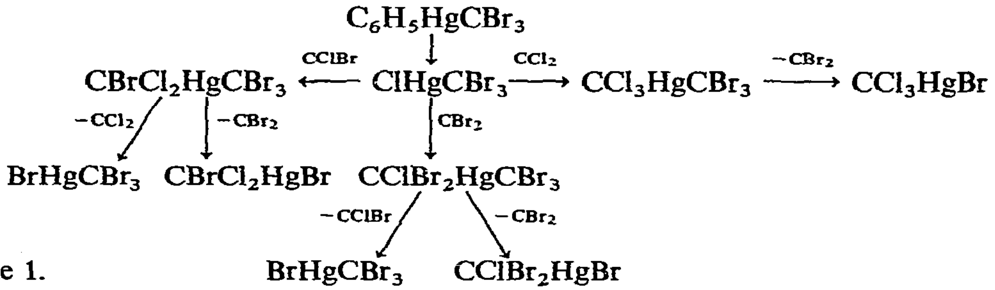
[a] Time during which excess of HCl was passed into the reaction mixture. [b] Theoretical yield of C_6H_5HgBr (7 mmoles), 2.51 g, m.p. 284–287°; theoretical yield of $ClHgCCl_2Br$, 2.79 g, m.p. 164–166°. [c] Yields of C_6H_6 and $CHCl_3$ were based on $C_6H_5HgCCl_2Br$.

The reaction of phenyl(trichloromethyl)mercury with hydrogen chloride in chlorobenzene solution at 85–88° gave mainly benzene (95%) and a small amount (0.2%) of chloroform. However, the study of the action of gaseous hydrogen chloride on phenyl(tribromomethyl)mercury produced results which were at first rather puzzling. For example, a reaction of this mercurial with HCl carried out in chlorobenzene at 85–87° gave benzene (77%) and the expected insertion product, $HCBr_2Cl$ (12.4%), but also formed were $HCCl_2Br$ (6.7%) and $HCCl_3$ (4.1%). No bromoform could be detected. Separate experiments showed that the chloroform and bromodichloromethane did not result from an exchange reaction (in the presence of phenylmercuric or mercuric halide) between $HCBr_2Cl$ and hydrogen chloride. A satisfactory explanation of the formation of $HCCl_2Br$ and $HCCl_3$ in these reactions was not available at the time we published a preliminary communication[2] concerned in part with these results. We have in the meantime achieved an understanding of these observations as a result of our finding that dihalocarbenes (as generated via

$C_6H_5HgCX_2Br$ compounds under neutral conditions) are capable of inserting into the Hg–Cl linkage.[7] It is to be noted that the benzene yield in the $C_6H_5HgCBr_3+HCl$ reaction was 80%. Thus the main reaction occurring was that shown in eqn. (5).

$$C_6H_5HgCBr_3+HCl \xrightarrow{80°} C_6H_6+ClHgCBr_3 \tag{5}$$

Insertion of CBr_2 into the Hg–Cl bond of $ClHgCBr_3$ would give $ClBr_2CHgCBr_3$, a compound which is a source both of CBr_2 and CBrCl. The latter could insert into an Hg–Cl linkage, giving an Hg–CCl_2Br compound, which in turn would be a source of CCl_2. Finally, insertion of CCl_2 thus formed into an Hg–Cl function would result in formation of an Hg–CCl_3 species. All of these reactions would be occurring in the presence of HCl, and thus the formation of $HCCl_2Br$ and $HCCl_3$ in low yield is explained by insertion of CClBr and CCl_2 into H–Cl. On the basis of these considerations, one would predict that the solids remaining upon completion of the reaction between $C_6H_5HgCBr_3$ and hydrogen chloride would be mostly $ClHgCBr_3$, together with smaller amounts of Br_3CHgBr, $ClBr_2CHgBr$, $BrCl_2CHgBr$ and Cl_3CHgBr (Scheme 1). These ideas have been verified by experiment.



Scheme 1.

Treatment of the solids from this $C_6H_5HgCBr_3+HCl$ reaction with excess of bromine in carbon tetrachloride (a semiquantitative analytical procedure for determination of trihalomethyl groups on mercury[8]) gave CBr_3Cl (10.4%), CBr_2Cl_2 (5.1%), and $CBrCl_3$ (9.8%), as well as large amounts of carbon tetrabromide. (The cleavage of pure $ClHgCBr_3$ and $C_6H_5HgCBr_3$ with bromine gave only carbon tetrabromide). This indicated that in addition to CBr_3Hg compounds there were present CBr_2ClHg, $CBrCl_2Hg$ and CCl_3Hg compounds. The latter are relatively stable and thus accumulate, so that the amount of CCl_3Br formed is larger than the amount of CCl_2Br_2. Another experiment in which 5 mmoles each of phenyl(tribromomethyl)mercury and pure tribromomethylmercuric chloride were allowed to react in benzene at 75–80° gave mercurial products, the brominolysis of which produced CBr_3Cl (21%), CBr_2Cl_2 (6%) and $CBrCl_3$ (6%). Thus the reactions shown in Scheme 1 do indeed occur*.

The effect of temperature on the reaction(s) occurring between phenyl(tribromomethyl)mercury and hydrogen chloride was studied (Table 2). Again, at

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* Our initial, tentative ideas[2] concerning the origin of the $HCCl_2Br$ and $HCCl_3$ thus are not correct. Also, the reactions used to show that this "Cl–Br exchange" occurred prior to haloform formation were confused by the occurrence of CBr_2 insertion into Hg–Cl bonds during the reaction of the solid mercurial product with cyclohexene in the presence of diphenylmercury.

lower temperatures C_6H_5–Hg cleavage occurs to the exclusion of the insertion of CX_2 into H–Cl.

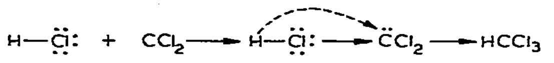
Qualitative experiments with $C_6H_5HgCClBr_2$ were carried out. At 80° the phenyl cleavage reaction predominated, but small amounts of $HCCl_2Br$ and lesser amounts of chloroform were formed when hydrogen chloride was bubbled into a solution of this mercurial.

The fact that in these reactions haloform formation is favored with increasing temperature and that maximum yields are obtained at temperatures at which CX_2 transfer reactions of $C_6H_5HgCX_2Br$ compounds are rapid[3] suggests that we are indeed dealing with CX_2 insertion into the H–Cl bond in the haloform-producing reaction. It is noteworthy that in the case of phenyl(trichloromethyl)mercury, whose CCl_2 transfer reactions at 80° are very much slower than those of the other three mercurials[3], phenyl–mercury cleavage occurred to the virtual exclusion of chloroform formation when its reaction with hydrogen chloride was carried out at 85–88°.

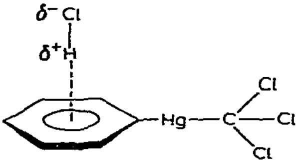
The benzene yields in these reactions also deserve further comment. The yields of benzene produced decreased as the temperature was raised. It appears that the C_6H_5–Hg bond in $C_6H_5HgCX_3$ compounds is cleaved rapidly by hydrogen chloride, but that phenylmercuric bromide is quite unreactive toward HCl under these conditions. This was confirmed in an experiment in which a suspension of 7 mmoles of phenylmercuric bromide in chlorobenzene at 80° was treated with hydrogen chloride for 30 min. Benzene was produced in only 6.5% yield and a 91% recovery of phenylmercuric bromide was realized. The lower yields of benzene in the $C_6H_5HgCX_2Br$ + HCl reactions at high temperature must be due primarily to the more rapid consumption of $C_6H_5HgCX_2Br$ in the insertion reaction at those temperatures and the fact that the insoluble phenylmercuric bromide produced thereafter is rather inert with respect to cleavage by HCl. Once phenyl cleavage has taken place, the resulting $ClHgCX_2Br$ species appear to be quite unreactive toward hydrogen chloride. No cleavage of CX_2Br by HCl occurs, and no CX_2 extrusion. The latter process appears to be more favorable once dihalocarbene insertion into the Hg–Cl bond of $ClHgCX_2Br$, giving a bis(trihalomethyl)mercury compound, has taken place. Phenyl cleavage is much more favorable in the case of $C_6H_5HgCBr_3$ than it is with $C_6H_5HgCCl_2Br$. This could be the result either of a more rapid C_6H_5–Hg cleavage process or of slower CBr_2 transfer to HCl.

Two main mechanisms can be suggested for the insertion of CX_2 into H–Cl: (*a*) nucleophilic attack by CX_2 (which is in the singlet state, according to all available evidence, and thus has a lone pair of electrons in an sp^2 orbital) at the proton (eqn. 6), or, (*b*) electrophilic attack by CX_2 (which also has a vacant $2p$ orbital) at chlorine, followed by proton migration from chlorine to carbon (eqn. 7). At present no clear no clear choice can be made between these possibilities, but we tend to favor the former alternative. It may be mentioned that $:CCl_2$ apparently reacts as a nucleophile with trialkylboranes[9].

$$Cl{-}H + {:}CCl_2 \rightarrow Cl^-[CCl_2H]^+ \rightarrow HCCl_3 \qquad (6)$$


(7)

In conclusion, the report by Nesmeyanov, Freĭdlina and Velichko[10] that cleavage of phenyl(trichloromethyl)mercury by methanolic HCl gives a quantitative yield of phenylmercuric chloride merits consideration, especially since, as we have shown, in chlorobenzene solution the products are benzene and CCl_3HgCl. We have confirmed the report of the Russian authors: cleavage of $C_6H_5HgCCl_3$ by one equivalent of concentrated, aqueous HCl diluted with methanol at room temperature gave phenylmercuric chloride in 80% yield, together with major amounts of chloroform and only a minor (4%) quantity of benzene. This marked solvent effect is of some interest and very likely is due to the different nature of hydrogen chloride in these solvent systems: covalent H–Cl molecules in chlorobenzene and ionized oxonium species, $[CH_3OH_2]^+Cl^-$ and $[H_3O]^+Cl^-$, in the aqueous methanol system. It is possible that in chlorobenzene attack at the phenyl ring is facilitated by hydrogen bonding between the H–Cl and the phenyl substituent on mercury (I). With the ionized oxonium species such hydrogen bonding is neither favorable nor necessary, and it may simply be a matter of which carbon atom attached to mercury is more liable to attack by a charged electrophile. In the present case, as one might expect, it is the highly electronegative CCl_3 group which is cleaved.



(I)

EXPERIMENTAL

General comments

All reactions were carried out under an atmosphere of prepurified nitrogen. Analyses were performed by Dr. S. M. Nagy, M.I.T. Microchemical Laboratory. Phenyl(trihalomethyl)mercury compounds were prepared as described in a previous part of this series[8]. Anhydrous hydrogen chloride was purchased from the Matheson Company.

Reaction of phenyl(bromodichloromethyl)mercury with hydrogen chloride

These reactions were carried out using a constant temperature bath capable of maintaining a temperature constant within 1–3°. One such reaction is described as an example of the procedure used.

Phenyl(bromodichloromethyl)mercury, 3.08 g (7 mmoles), was placed in a 50-ml three-necked flask equipped with a thermometer, a condenser, a magnetic stirring unit and a gas inlet tube. Thirty ml of dry chlorobenzene was added. The contents were heated to 85–88° and then gaseous hydrogen chloride was passed into the clear solution while stirring vigorously. Precipitation of solid material occurred within 3 min. The reaction was discontinued after HCl had been passed in for 15 min. Solid material, 2.57 g, m.p. 275–277° (turned brown), was isolated. The filtrate was trap-to-trap distilled at 0.05 mm with pot temperature to 80°. The distillate was analyzed by glpc at jacket temperature 90°, 15 psi helium with a 7 ft. glass 25% SE-30 on Chromosorb P analytical column using an M.I.T. isothermal gas chromatograph, with toluene as internal standard. The results of these experiments are given in Table 1.

Reaction of phenyl(tribromomethyl)mercury with hydrogen chloride

Essentially the same procedure as that described above was used. In a typical example, phenyl(tribromomethyl)mercury (5.29 g, 10 mmoles) was placed in the reaction flask containing 20 ml of chlorobenzene under an atmosphere of nitrogen. The contents were heated to 85°. Hydrogen chloride was bubbled into the vigorously stirred solution. White solid precipitated within one min; passage of HCl was discontinued after 20 min at 85–87°. Solid residue, 3.23 g, m.p. 148–151° with slow decomposition (and with a portion not melted at 200°), was obtained. The filtrate was trap-to-trap distilled at 0.01 mm. The residue, slightly yellow solid, 1.24 g, m.p. 138–142°(dec.), was impure CBr_3HgCl. The combined residues (4.47 g) were saved for the brominolysis experiment.

The clear distillate was analyzed by glpc (25% General Electric SE-30 on Chromosorb P, 85° jacket temperature, 14 psi helium, toluene internal standard) and shown to contain benzene (77%), chloroform (4.1%), bromodichloromethane (6.7%) and dibromochloromethane (12.4%). The products were identified by comparison of their glpc retention times and their infrared spectra with those of authentic samples. Four other reactions were carried out at lower temperatures. The results are given in Table 2.

TABLE 2

REACTION OF $C_6H_5HgCBr_3$ WITH HCl IN CHLOROBENZENE

Temp. range (°C)	*C_6H_6 yield (%)*	*$CHClBr_2$ yield (%)*	*$CHCl_2Br$ yield (%)*	*$CHCl_3$ yield (%)*	*Reaction time (min)*
85–87	77 (80)[a]	12.4 (12.0)	6.7 (7.5)	4.1 (5.1)	20
73–75	85	3.4	0.7	0.7	20
58–59	94	0	0	0	20[b]
30–32	95	0	0	0	30[b]
4–7	94	0	0	0	50[b]

[a] Duplicate run values in parentheses. [b] A quantitative yield of $ClHgCBr_3$, m.p. 147–148° (dec.), was obtained.

The solids produced in this reaction (4.47 g) were treated with 11 ml of 1 *M* bromine in carbon tetrachloride at room temperature for 90 min. Subsequently 1.5 g of anhydrous magnesium sulfate and 3.0 g of powdered sodium thiosulfate pentahydrate were added. Stirring was continued until the bromine color had been discharged. The mixture was distilled in vacuum into a trap at −78°. The distillate was dried with $MgSO_4$. Glpc analysis of the distillate [20% SE-30 on Chromosorb W, F & M Model 700 temperature programmed (90–180° at 3° per min.) gas chromatograph] showed the presence of $CClBr_3$ (10.4%), CCl_2Br_2 (5.1%) and CCl_3Br (9.8%).

Reaction of phenyl(tribromomethyl)mercury with tribromomethylmercuric chloride

A dry three-necked flask equipped with a dropping funnel, reflux condenser topped with a nitrogen inlet tube, and a magnetic stirring assembly, was charged with

2.65 g (5 mmoles) of $C_6H_5HgCBr_3$, 2.44 g (5 mmoles) of CBr_3HgCl and 13 ml of dry benzene under an atmosphere of prepurified nitrogen. The reaction mixture was stirred and heated at 75–80° for 4 h. Subsequently 17 ml of 1 *M* bromine in carbon tetrachloride was added at room temperature over a 15 min period. Further workup as in the experiment described above established that $CClBr_3$ (20.6%), CCl_2Br_2 (5.9%) and CCl_3Br (5.8%) had been formed, in addition to carbon tetrabromide and bromobenzene.

In a reaction carried out between these mercurials at 40° for 3 h only $CClBr_2Hg$ compounds appeared to have been formed, since brominolysis gave only $CClBr_3$ (9.1%).

Reaction of phenylmercuric bromide with hydrogen chloride

Phenylmercuric bromide, 2.5 g (m.p. 284–287°), and 30 ml of chlorobenzene were heated to 70–80° in the reaction flask. Hydrogen chloride gas was bubbled into the vigorously stirred reaction mixture for 30 min. Unreacted phenylmercuric bromide was filtered, washed and dried; 2.27 g (91%) of phenylmercuric bromide, m.p. 284–287°, was recovered. Analysis of the filtrate (same glpc conditions as before) established that benzene had been formed in 6.5% yield.

Preparation of trihalomethylmercuric chlorides

Trichloromethyl-, bromodichloromethyl-, dibromochloromethyl- and tribromomethylmercuric chloride were prepared by treatment of a chlorobenzene solution of the respective phenyl(trihalomethyl)mercury compound with anhydrous hydrogen chloride at 23°. The reactions were carried out on a 10 mmole scale. The benzene yields were determined by glpc and were found to be 93% or above.

CCl_3HgCl. 91% yield; m.p. 190–191° (lit.[11] m.p. 193–194°). (Found: C, 3.46; Cl, 39.26; CCl_4Hg calcd.: C, 3.39; Cl, 40.01%.)

$CCl_2BrHgCl$. 95% yield; m.p. 164–166° (slow dec.). (Found: C, 3.29; Br, 19.95; Cl, 26.90; Hg, 50.07; $CBrCl_3Hg$ calcd.: C, 3.07; Br, 20.02; Cl, 26.66; Hg, 50.28%.)

$CClBr_2HgCl$. 96% yield; m.p. 155–156° (slow dec.). (Found: C, 2.79; Br+Cl, 51.64; CBr_2Cl_2Hg calcd.: C, 2.71; Br+Cl, 52.02%.)

CBr_3HgCl. 96% yield; m.p. 148–150° (dec.). (Found: C, 2.52; Br+Cl, 56.13; CBr_3ClHg calcd.: C, 2.46; Br+Cl, 56.42%.)

All the CX_3HgCl compounds were recrystallized from benzene. They separated out as fine needle-like, crystalline solids on cooling.

Reaction of phenyl(trichloromethyl)mercury with HCl in aqueous methanol

Phenyl(trichloromethyl)mercury, 3.49 g (8.8 mmoles), was placed in a 100-ml round-bottomed flask. A solution of about 8.8 mmoles of HCl, made up by diluting 12 *M* hydrochloric acid with methanol to *ca.* 15 ml total was added. The reaction mixture was shaken gently for 30 min. White, crystalline solid formed almost immediately, and the needle-like starting material gradually disappeared as the latter formed. The solid product was filtered to give 1.84 g of material, m.p. 259–260°, which was identified by m.p. and mixture m.p. as phenylmercuric chloride. The filtrate was distilled in vacuum into a receiver at −78°. The solid residue was washed with benzene to leave 0.42 g of phenylmercuric chloride, m.p. 258–260°, to give a total yield of 2.26 g (80%). The benzene washings were evaporated, leaving 0.7 g of solid, melting range 120–140° (dec.).

The distillate was analyzed by glpc using toluene as internal standard. Benzene (4%) and chloroform (65%) were identified.

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SUMMARY

Dihalocarbenes (CCl_2, CClBr, CBr_2) obtained from the corresponding $C_6H_5HgCXYBr$ compounds have been found to insert into the H–Cl bond, giving haloforms, $HCCl_3$, $HCCl_2Br$ and $HCBr_2Cl$, respectively. In chlorobenzene solution at 80° C_6H_5–Hg cleavage is a competing reaction which in the case of $C_6H_5HgCBr_3$ accounts for 80% of the HCl consumed. The resulting CBr_3HgCl introduces further complications, since CX_2 insertion into the Hg–Cl bond occurs, giving $HgCX_2Br$ compounds which are sources of carbenes other than those derived from the starting mercurial. At room temperature only the C_6H_5–Hg cleavage reaction is observed, and this allowed preparation of pure $ClHgCCl_3$, $ClHgCCl_2Br$, $ClHgCClBr_2$ and $ClHgCBr_3$. The action of methanolic HCl on $C_6H_5HgCCl_3$ produces phenylmercuric chloride and chloroform; a possible explanation for this marked solvent effect is presented.

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