Journal of Organometallic Chemistry, 160 (1978) 7—16

REACTIONS OF DIASTEREOMERIC L-MENTHYL ESTERS OF α-MERCURATED PHENYLACETIC ACIDS WITH TRIS(TRIPHENYLPHOSPHINE)PLATINUM. STEREOCHEMISTRY AND MECHANISM OF THE PLATINUM CARBENOID $(Ph_3P)_2Pt^0$ INSERTION INTO THE CARBON—MERCURY BOND *

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(Received June 20th, 1978)

Summary

The reaction between $(Ph_3P)_3Pt^0$ and diastereoisomeric L-menthyl esters of α-bromomercuriphenylacetic acid or related R_2Hg compounds has been investigated. The preparation of σ-organomercuryplatinum and corresponding σ-organoplatinum derivatives is reported. Diastereomer RHgBr (I), $[\alpha]_D^{20}$ —120°, and diastereomer II, $[\alpha]_D^{20}$ —18°, afforded σ-organoplatinums having $[\alpha]_D^{20}$ —21.9° and —17.5° respectively. This suggests that organomercurial salts undergo the L_2Pt insertion into both Hg—Br and Hg—C bonds, the latter process being a stereoselective one. It can be deduced from the values of the optical rotations that the L_2Pt insertion into the C—Hg bond occurs with retention of configuration. Different mechanisms for this reaction are discussed. The novel cleavage of the Pt—Hg bond with bromoform is also described.

Introduction

The tendency for the increase in the oxidation state of the metal is characteristic of zerovalent d^{10} complexes of platinum, palladium and nickel. These reactions are known as oxidative addition and are mostly investigated for acids and organic halides. Recently we have found a related reaction of Pt^0 and Pd^0 complexes with organomercurials RHgX and R_2Hg [1—3] that appeared to be a general pattern for the reactivity of the organometallics of different non-transition metals [4].

$$R_nM + L_3Pt \rightarrow R_{n-1}M—L_2Pt—R + L$$

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* Dedicated to Professor Ernst Otto Fischer on the occasion of his 60th birthday on November 10, 1978.

For M = Hg, the primary products having a Hg—Pt(Pd) bond are usually unstable and decompose readily under heat or light to afford σ-organotransition metals. This route is a useful preparative procedure. Remarkably, the L_2Pt insertion may occur not only into a M—Hal bond which can be considered as a common oxidative addition of organic halides but into a M—C bond as well, for example, in R_2Hg. Furthermore, the reaction with R_3SnHal was proved to involve the Sn—C bond rather than Sn—Hal bond [5].

The carbenoid-type reactivity of zerovalent platinum complexes has been recognised some years ago [6]. The electron-releasing character of phosphine ligands and a particular readiness of polyfluoroorganomercurials to react with $(R_3P)_2Pt^0$ allowed the proposition that $(Ph_3P)_nM^0$ complexes behave as nucleophilic carbenoids [3,7]. On the other hand, dibenzylideneacetone complexes of Pt^0 and Pd^0 [8] which can be expected to provide slightly electrophilic carbenoids also undergo similar reactions with organomercurials [8]. However, the presence of very strong electron-withdrawing ligands like PF_3 is known to prevent completely the oxidative addition of acids and organic halides [9].

All reactions leading from M^0 to M^{+2} complexes within the same triad of metals should be considered together. The mechanisms of oxidative addition of organic halides have been studied but conclusions are contradictory. No definite choice could be made generally between homolytic and heterolytic pathways [10]. The most useful mechanistic information can be obtained from stereochemical studies. Recently Stille et al. [10,11] have shown that the oxidative addition of optically active benzyl-α-d halides to $(Ph_3P)_4Pd$ proceeds with the predominant inversion of configuration of carbon. A similar reaction with $(Ph_3P)_4Ni$ has been reported [12] to result in nearly complete racemization. Optically active 8-(α-bromoethyl)quinoline reacts with $(Ph_3P)_3Pt$ to form a chelated organoplatinum product with net inversion of configuration at carbon [13].

The main aim and choice of model

The structure of organomercurials suitable for the stereochemical study of the L_2Pt insertion should meet some important requirements. First of all, the demercuration step which is evidently homolytic must be excluded or inessential for the final conclusion. Generally, R_2Hg is more suitable than RHgX since the insertion is undergone more readily into the Hg—Hal than the Hg—C bond. Certainly, the metal should be attached to a chiral carbon atom in an optically active molecule.

To summarize, the following requirements can be indicated for an ideal model:

1. A dialkylmercury R_2Hg is needed which has a metal atom at the chiral centre.
2. The organomercuryplatinum intermediate must be stable enough to be isolated but may be cleaved selectively at the Hg—Pt bond under mild conditions.
3. The final σ-alkylplatinum product should be chemically and configurationally stable.
4. There must be the opportunity to correlate the configurations of starting organomercurial and resulting organoplatinum derivatives.

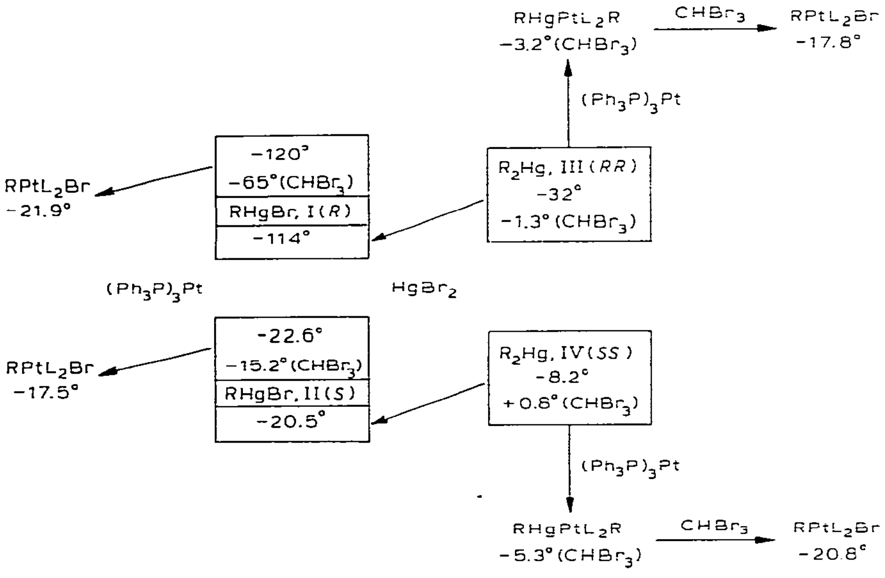
At the present time only a paucity of optically active molecules with the chiral carbon-bearing mercury is known. Further, one should take into account that alkylplatinums possessing a β-hydrogen tend to undergo β-elimination to afford hydrides L_2PtHX. So we have chosen as a model the well-known L-menthyl esters of α-mercuribis(phenyl)acetic acid [14]. They are diastereomers which certainly brings some complications but the related enantiomeric ethyl esters were obtained with difficulty only as salts RHgBr, not R_2Hg [15].

The isolation of diastereomeric L-menthyl esters of α-bromomercuriphenylacetic acid I and II, their symmetrization and reverse reaction were re-investigated [16]. The diastereomeric purity of R_2Hg was estimated on the basis of the reaction with mercuric bromide in THF which is an S_E2 reaction which proceeds with complete retention of configuration.

$$R_2Hg + HgBr_2 \rightarrow 2\ RHgBr$$

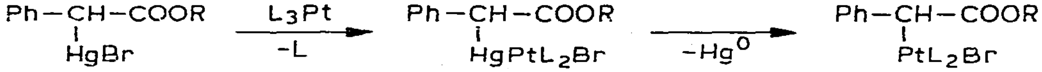
Pure diastereomer RHgBr, I, which has the *R*-configuration at the carbon bearing the mercury atom exhibits $[\alpha]_D^{20}$ —132° (C_6H_6) and diastereomer II, nearly 90% pure, has $[\alpha]_D^{20}$ —18° (*S*-configuration). The related dialkylmercurials III and IV were obtained with $[\alpha]_D^{20}$ —32° and —8.2° respectively. Chemical reactions and stereochemical correlations are shown in Scheme 1.

SCHEME 1. When solvent not specified, optical rotation measured in benzene.

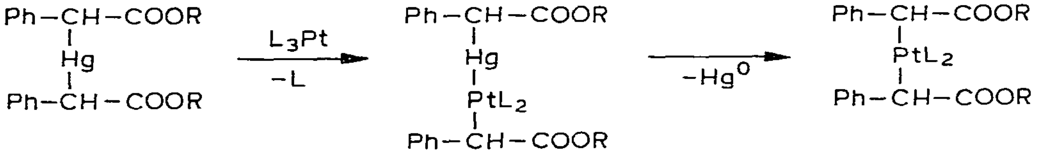


Synthesis of σ-platinum derivatives or phenylacetic esters

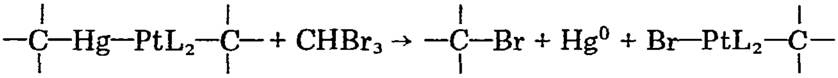
Reaction between the esters of α-bromomercuriphenylacetic acid and $(Ph_3P)_3$-Pt gives rise easily to mercuryplatinum compounds which on decomposition give α-bromobis(triphenylphosphine)platinum phenylacetic esters. Preparatively, this reaction may be carried out in high yield even without isolation of the bimetallic intermediate. The presence of a good-solvating solvent like THF and daylight favours the mercury elimination in situ.



It is remarkable that oxidative addition of esters of α-bromophenylacetic acid to $(Ph_3P)_3Pt$ fails to give σ-alkylplatinums, L_2PtBr_2 being the sole product of the reaction. This finding confirmed once more the preparative value of the organomercurial route compared to the halide oxidative addition which has been clearly demonstrated before by the synthesis of cycloalkylplatinums [17]. Also symmetrical organomercurials R_2Hg afford smoothly bimetallic and then platinum derivatives as previously reported [17].



In this work, we have found a novel mild method to cleave selectively the Pt—Hg bond with bromoform. Metallic mercury, the corresponding alkylplatinum and alkyl bromide are formed.

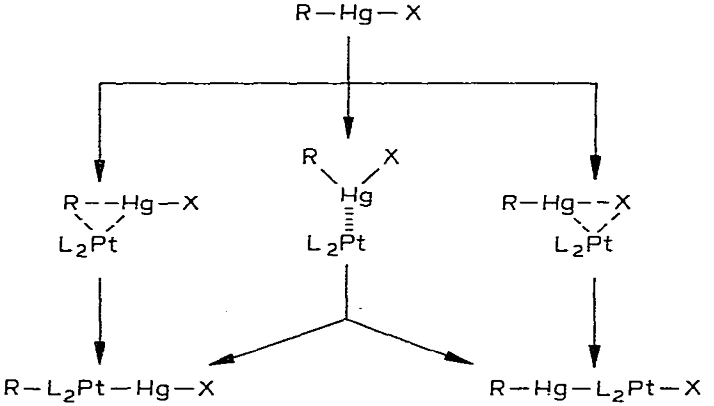


$$-\overset{|}{\underset{|}{C}}-Hg-PtL_2-\overset{|}{\underset{|}{C}}- + CHBr_3 \rightarrow -\overset{|}{\underset{|}{C}}-Br + Hg^0 + Br-PtL_2-\overset{|}{\underset{|}{C}}-$$

Possible mechanisms for insertion

A priori different mechanisms can be expected for the L_2Pt insertion into a mercury—element bond. Their realization for a particular case will depend on the nature of both groups attached to the metal. The inversion of configuration at carbon for this kind of reaction seems to be improbable since the RHg— group has, unlike halogen, no propensity to leave as anion in an S_N2-type process. Reaction pathways without the complete loss of stereochemical configuration may be divided into two groups. The initial attack by L_2Pt carbenoid may occur

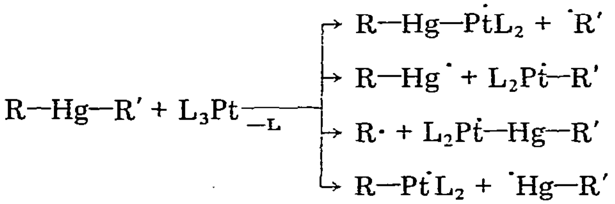
SCHEME 2



either at the mercury—element bond to give a three-membered transition state or at the mercury atom. In this latter case, the ylide-like species is formed which undergoes migration of one group from mercury to platinum. Actually, these two possibilities can hardly be distinguished since the expected stereochemical result would be retention of configuration (Scheme 2).

If X = Hal, the reaction may be conveniently described as nucleophilic displacement at mercury $S_N2(Hg)$. The formation of a three-membered intermediate was discussed recently when we established the *cis*-arrangement of phosphine ligands in the X-ray study of $CF_3Hg(Ph_3P)_2PtCF_3$ [18].

Homolytic free-radical mechanisms are also possible. They will doubtless result in a complete loss of stereochemical configuration. In our case, free-radical synthesis of starting organomercurials affords an equilibrated mixture of I and II. Let us consider in detail different radical pathways.



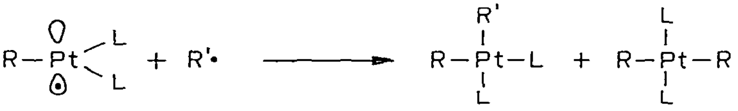
$$R{-}Hg{-}R' + L_3Pt \xrightarrow{-L} R{-}Hg{-}\dot{Pt}L_2 + \dot{R}' \qquad (1)$$

$$\rightarrow R{-}Hg^{\bullet} + L_2\dot{Pt}{-}R' \qquad (2)$$

$$\rightarrow R^{\bullet} + L_2\dot{Pt}{-}Hg{-}R' \qquad (3)$$

$$\rightarrow R{-}\dot{Pt}L_2 + {}^{\bullet}Hg{-}R' \qquad (4)$$

Present knowledge of RHg· species leaves little doubt that they loose mercury very rapidly. The intervention of this radical contradicts the formation of mercuryplatinum compounds. Among the homolytic mechanisms (eq. 1) is most probable, involving "carbon" and "platinum" radicals. The latter, $R\dot{Pt}L_2$, which has an electron localized mostly on three-coordinated platinum is evidently formed in the course of demercuration of mercuryplatinum compounds *. This radical may be thought to be planar and can be attacked either from in the plane or from outside it to give generally both isomeric *cis* and *trans* complexes of Pt^{II}:



Stereochemical results and mechanistic considerations

Each of the RHgX diastereomers has been treated with an equimolar quantity of zerovalent platinum complex in benzene/THF mixture. After metallic mercury had separated completely the solution was filtered and evaporated to dryness. The entire residue was taken for the optical rotation measurement, and the weight was corrected for triphenylphosphine present **. Experiments with I and II were performed using the same batches of Pt^0 complex and solvent. The

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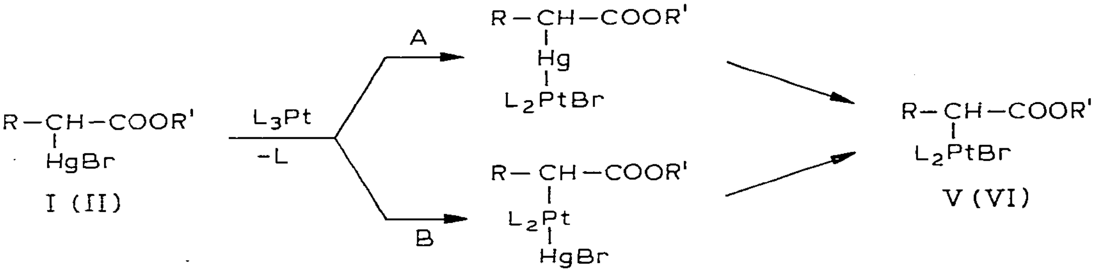
* A related Pt radical but containing the four-coordinated metal has been reported by Clark et al. [19].

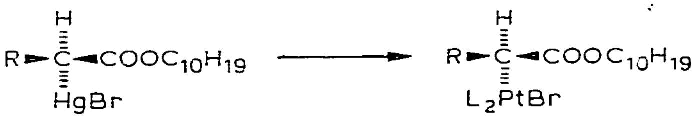
** Starting with L_3Pt complex which was usually obtained under our conditions one must expect 1 eq. of free phosphine liberated and with L_4Pt 2 eq. Substracting from the weighed amount the weight which corresponds 1.5 eq. involves an error ±7%.

specific rotation of V and VI appeared to be different: V obtained from I exhibited $[\alpha]_D^{20}$ −21.9 ± 1.5° and VI obtained from II had $[\alpha]_D^{20}$ −17.5 ± 1.2°. This result is in a good accord with the simultaneous insertion of L_2Pt into both Hg—Br (path A) and Hg—C (path B) bonds.



There is no doubt that path A dominates. The crystalline mercuryplatinum compound isolated from toluene solution, according to our preliminary X-ray study, has the bond series C—Hg—Pt—Br. Demercuration of this intermediate involves a "carbon" radical and hence does not retain configuration. Path B involves stereochemical control on the first stage while demercuration goes through a "platinum" radical and cannot influence the net sterochemical result. It is reasonable to assume that the entire stereoselectivity observed in this reaction comes from the contribution of path B.

With structural identity of I and II, one may expect the same ratio A/B under the same conditions. In this case, the average value $[\alpha]_D^{20}$ −19.7° has to reflect the specific rotation of the equilibrium mixture of V and VI. The change is due to one diastereomer being in excess. It is of positive sign for VI and of negative sign for V. It is impossible to make a quantitative estimation of the A/B ratio without knowing the absolute rotation of the organoplatinum diastereomers. However, one may affirm that *R*(−)-I affords *R*(−)-V and *S*(+)-II affords *S*(+)-VI. Following Brewster's concept of atomic asymmetry [20] replacement of mercury by platinum at the chiral centre of the same absolute configuration does not change the sign of optical rotation. Hence it follows that the chiral carbon bearing platinum has *R*-configuration in V and *S*-configuration in VI. Thus the sense of stereoselectivity in the course of L_2Pt insertion into the C—Hg bond should be net retention of configuration.

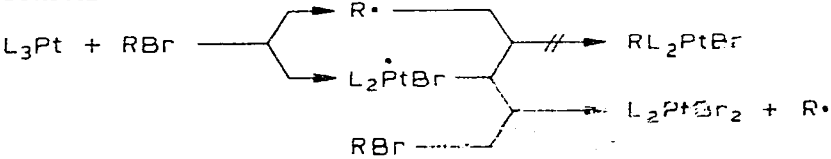


S (+) II

S (+) VI

It is of some interest why the organomercurial route affords successfully the organoplatinum product while the oxidative addition of the related alkyl bromide

SCHEME 3



fails. Apparently this fact is due to the high stability of the "carbon" radical $Ph\dot{C}HCOOR$ and the related capacity of this bromide to serve as a trap for $L_2\dot{P}tBr$. The reactions shown in Scheme 3 are likely to take place in this system.

It is worth emphasizing that the "carbon" radical in question has extremely high stability. The direct synthesis of organomercurials is known to fail with both benzyl bromide and bromoacetic esters under free-radical conditions. In this connection, it is not surprising that α-bromophenylacetic esters react with Pt^0 complexes in a homolytic way to produce L_2PtBr_2 instead of $AlkPtL_2Br$. With benzyl bromides giving less stable radicals, heterolytic mechanisms are realized [10,13]. All the more remarkable is that the organomercurial method gives good results even in this case which is the most favourable one for the radical pathway. Evidently, the bimetallic intermediate forms in a heterolytic way from the reactants and then undergoes homolysis of Hg—Pt bond to afford radicals $R\dot{P}tL_2$ and $RHg^\cdot$; the latter produces the alkyl radical $R^\cdot$ and the two radicals couple freely since there is no competition with alkyl bromide.

Structurally symmetrical dialkylmercury molecules unlike RHgX can undergo insertion in a single manner. We have prepared mercuryplatinum derivatives IX and X from diastereomers III and IV. Each of these crystalline compounds underwent cleavage of the Hg—Pt bond on treatment with bromoform. Using cold bromoform equilibrated with its solid phase, we were able to measure the optical rotation of IX and X before the decomposition. The measurement took not more than 2 min after which period separation of mercury from solution started. The specific rotations appeared to be different for IX and X, as shown in Scheme 1. For comparison the influence of bromoform on the rotation of organomercurials was also studied.

The cleavage of the Hg—Pt bond retains in the molecule just that chiral carbon configuration of which reflects the stereochemistry of the insertion stage. Unfortunately, the bromoform reaction can further destroy alkylplatinum to give the inorganic complex L_2PtBr_2. The isolable yield of $RPtL_2Br$ is in the range 30—40%. Since this further reaction can change the ratio of the diastereomers reliable determination of the stereochemistry of the insertion appears to be impossible.

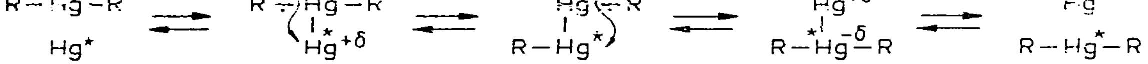
$$R{-}Hg{-}PtL_2{-}R \xrightarrow[\substack{-RBr\\-Hg}]{CHBr_3} Br{-}PtL_2{-}R \xrightarrow[-RBr]{CHBR_3} Br{-}PtL_2{-}Br$$

On the other hand, the direct elimination of mercury produces the dialkylplatinum generally as three diastereomers: RRL_2, RSL_2, SSL_2 (L stands in general for a menthyl group as a whole). The investigation of this reaction is in progress.

We wish here to note the analogy between the reaction under consideration and the exchange of organomercurials and metallic mercury which has been known for years [21].

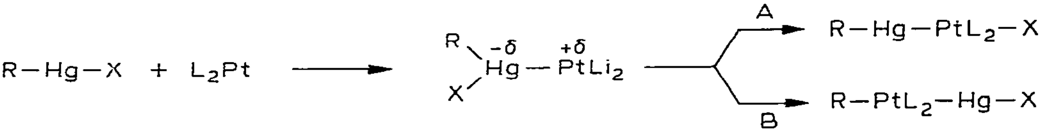
$$R{-}Hg{-}R + \overset{\star}{Hg} \rightleftharpoons R{-}\overset{\star}{Hg}{-}R + Hg$$

The investigation resulted in the following mechanism:



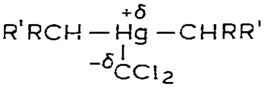
According to this mechanism [22,23], the key step is the formation of "organic calomel" which has an unsymmetrical, ylide-like structure. The "outside" mercury atom should play the role of electron donor and the "central" one a role of acceptor after rearrangement to a chain occurs.

Previously, we had an opportunity to note the resemblance between zerovalent complexes of platinum metals and free atoms of non-transition metals [24], for example, $(R_3P)_2Pt^0$ and Hg^0. The electron-donating ability of $(R_3P)_2Pt^0$ is not less than that of Hg^0. Therefore the formation of a bimetallic ylide intermediate is not less probable. The rearrangement may follow two pathways A and B if they are distinguishable. Thus, it is possible that both products have a common precursor and their ratio is the consequence of the difference in the aptitude of two ligands at mercury for migration.



Taking into account the charge separation within the ylide intermediate it is natural that the more nucleophilic halogen undergoes easier intramolecular migration compared to the alkyl group. This supposition confirms that the reaction will proceed more readily if electron-withdrawing groups are present. It also suggests the retention of carbon configuration in the course of the intramolecular shift of the group R.

To our knowledge, a unique example of a study of the stereochemistry of the carbenoid insertion into a C—Hg bond has been previously described by Landgrebe and Mathis [25]. They have studied the reaction of optically active di-sec-butylmercury with dichlorocarbene generated from CCl_3COOEt and NaOMe. The product was converted into the known (+) α-methylbutyric acid. It was found that full retention of configuration took place. The authors [25] considered as possible two mechanisms: a concerted attack on the C—Hg bond and the formation of an ylide-like intermediate.



$$\mathrm{R'RCH-\underset{{}^{-\delta}CCl_2}{\overset{+\delta}{Hg}}-CHRR'}$$

Since CCl_2 is an electrophilic carbene, the charge distribution in this species should be opposite to that expected for Hg—Hg or Hg—Pt ylides. However, the exchange of phosphine ligands at platinum for an accepting ligand like DBA may essentially alter the electronic structure of the intermediate.

Experimental

All experiments with Pt^0 complexes were performed under argon. Optical rotation was measured using a Perkin—Elmer 241 instrument at 20°C, in benzene if no other solvent is indicated. *c* normally was in the range 0.5—5.0 g/100 ml; the length of the polarimetric tube 2 or 1 cm.

Reaction of L-menthyl ester of α-bromophenylacetic acid with $(Ph_3P)_3Pt$. 0.7 g of α-bromo ester, $[\alpha]_D^{20}$ + 10.6° or —84.2°, was treated in 20 ml of benzene with

0.5 g of L_3Pt. After mixing the reactants the colour faded immediately and a white solid separated. This was washed with hexane and dried. Yield of L_2PtBr_2 was 0.4 g (80%), m.p. 269°C.

Reaction of α-bromoester with Ph_3P. 0.3 g of α-bromo ester, $[\alpha]_D^{20}$ +10.6°, in 3 ml of benzene was treated with 0.08 g of Ph_3P. The observed rotation in a 1 cm tube was α_D +0.128°. After 24 h it attained the value α_D —0.090° which corresponds to $[\alpha]_D^{20}$ —7.1° calculated for a quaternary phosphinium salt.

Synthesis of mercuryplatinum derivatives

1. *From RHgBr, diastereomer II, $[\alpha]_D^{20}$ —20.2°.* To a solution of 0.154 g of II in 9 ml of THF was added 0.28 g of $(Ph_3P)_3Pt$ in 20 ml of toluene. The reaction mixture was maintained at —6° in the dark for 48 h. The product partly separated as olivegreen crystals. 50 ml of cold hexane was added to complete the separation. After the usual work-up 0.23 g (72%) of dry product was obtained, m.p. about 135°C (dec.); IR: ν(C=O) 1705 cm^{-1} (KBr). Found: C, 52.65; H, 4.45; Br, 6.88; Hg, 15.48. $C_{54}H_{55}BrHgO_2P_2Pt \cdot 0.5\ C_6H_5CH_3$ calcd.: C, 52.35; H, 4.47; Br, 6.08; Hg, 15.17%.

2. *From RHgBr, diastereomer I, $[\alpha]_D^{20}$ —120°.* Similarly 0.070 g of I and 0.130 g of $(Ph_3P)_3Pt$ afforded 0.133 g (74%) of the product which decomposed at ca. 129°C. Elemental analysis was obtained for a sample from the sample having $[\alpha]_D^{20}$ —105°. Found: C, 53.26; H, 4.39; Br, 5.58; Hg, 14.12%.

3. *From R_2Hg, diastereomer IV, $[\alpha]_D^{20}$ —8.2°.* 0.24 g of IV in 4 ml of THF was treated with 0.34 g of $(Ph_3P)_3Pt$ in 13 ml of toluene. The colour of the solution turned from yellow to orange. After 48 h at —13°C the green-yellow product crystallized partly. 50 ml of cold hexane was added and the precipitate X collected after a further 24 h. Yield 0.233 g (67%), m.p. 106°C (dec.), $[\alpha]_D$ —5.3° (*c* 9, $CHBr_3$). IR: ν(C=O) 1715 cm^{-1} (KBr). Found: C, 59.63; H, 5.43; Hg, 14.02. $C_{72}H_{80}O_4P_2PtHg$ calcd.: C, 59.14; H, 5.47; Hg, 13.69%.

4. *From R_2Hg, diastereomer III, $[\alpha]_D^{20}$ —32°.* Similarly a mercuryplatinum derivative IX was prepared in 79% yield, m.p. 102°C (dec.), $[\alpha]_D$ —3.2° (*c* 9, $CHBr_3$).

Demercuration of mercuryplatinum derivatives

5. 0.13 g of the Hg—Pt compound prepared from I was treated with a mixture THF/toluene 1/1. The suspension was exposed to daylight with stirring until the bimetallic derivative had decomposed completely. Mercury was removed, THF evaporated in vacuo and the light-yellow product was precipitated with 50 ml of hexane. The yield of alkylplatinum was 0.071 g (65%), m.p. 139—140°C (dec.). IR: ν(C=O) 1715 cm^{-1} (KBr). Found: C, 61.06; H, 5.22; Br, 7.46. $C_{54}H_{55}BrO_2P_2Pt$ calcd.: C, 60.25; H, 5.13; Br, 7.45%.

Similarly, platinum derivatives from the decomposition of other mercuryplatinum compounds described above were obtained in 75% yield. Satisfactory analytical data were found.

Direct preparation of platinum derivatives without isolation of bimetallic intermediates

6. *Alkylbis(triphenylphosphine)platinumbromides.* 0.17 g of either I or II in 8 ml of THF was treated with 0.32 g of $(Ph_3P)_3Pt$ in 17 ml of benzene. The

reaction mixture was exposed to daylight until mercury separation was complete. From the filtrate THF was evaporated and the product was precipitated with hexane. The mixture of alkylplatinum diastereomers was obtained in ca. 70% yield. IR: ν(C=O) 1730—1735 cm^{-1} (KBr). Found: C, 60.25; H, 5.13; Br, 7.45. $C_{54}H_{55}BrO_2P_2Pt$ calcd.: C, 59.68; H, 4.96; Br, 7.83%.

7. *Dialkylbis(triphenylphosphine)platinum.* 0.37 g of either III or IV in 5 ml of THF was treated with 0.5 g of $(Ph_3P)_3Pt$ in 20 ml of toluene. The orange solution was maintained in daylight until mercury separation was complete. The usual work-up gave about 80% of R_2PtL_2. IR: ν(C=O) 1725 cm^{-1} (CH_2Cl_2). Found: C, 68.31; H, 6.32. $C_{72}H_{80}O_4P_2Pt$ calcd.: C, 67.59; H, 6.17%.

Cleavage of dialkylmercuryplatinum compounds with bromoform

8. 0.46 g of R_2HgPtL_2 (IX), $[\alpha]_D^{20}$ —3.2° (*c* 9, $CHBr_3$), was dissolved in 5 ml of cold bromoform. As soon as the separation of mercury stopped (about 15 min) 50 ml of cold hexane was added to afford 0.31 g of yellow solid, m.p. 114—204°C (dec.) which was extracted with benzene (5 × 10 ml) leaving L_2PtBr_2. From benzene 0.120 g (32%) of alkylplatinum was isolated, m.p. 136—137°C (dec.), $[\alpha]_D^{20}$ —18°. IR: ν(C=O) 1708 cm^{-1} (KBr). Found: C, 59.64; H, 4.36; Br, 7.65. $C_{54}H_{55}BrO_2P_2Pt$ calcd.: C, 60.25; H, 5.13; Br, 7.45%. Similarly R_2HgPtL_2 (X), $[\alpha]_D$ —5.3° (*c* 9, $CHBr_3$), gave 27% of $RPtL_2Br$, m.p. 127—134°C (dec.), $[\alpha]_D^{20}$ —20.8°. IR: ν(C=O) 1704 cm^{-1} (KBr). Found: C, 58.96; H, 4.70; Br, 8.34%.

Acknowledgement

Dr. L.L. Troitskaya is much acknowledged for valuable discussions in the course of this work.

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