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A New Route to Mercury-Containing Triphenylphosphonium Salts with Electron-Withdrawing Groups at the P Atom and Some Transformations of These Compounds

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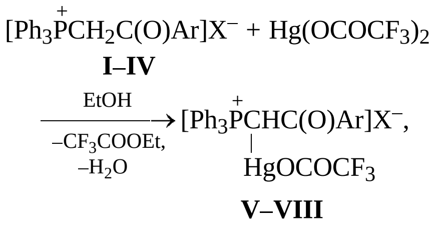
Abstract—(Aroylmethyl)triphenylphosphonium salts react with mercury(II) trifluoroacetate with replacement of the labile methylene hydrogen atom by the $HgOCOCF_3$ group to give mercury-containing triphenylphosphonium salts in quantitative yields. Treatment of the mercurated salts with potassium carbonate yields mercury-containing triphenylphosphonium ylides of symmetrical and unsymmetrical structures, which enter Wittig reaction with aromatic aldehydes to form the corresponding mercurated olefins (*Z* isomers) and triphenylphosphine oxide.

Mercury-containing triphenylphosphonium salts prepared for the first time more than 40 years ago [1] attract researchers' attention thanks to their potential in organic synthesis [2]. These compounds were prepared by addition of mercury salts to stable [3–7] and unstable [1, 8] triphenylphosphorylides; in the latter case, however, difficult-to-separate mixtures of products were obtained.

In this work, proceeding with studies on mercuration of onium compounds containing fairly labile hydrogen atoms [9], we performed for the first time the similar reactions with a number of accessible and stable triphenylphosphonium salts [10, 11]. We found that refluxing of equimolar amounts of mercury(II) trifluoroacetate and (aroylmethyl)triphenylphoshonium salts **I**–**IV** in anhydrous ethanol results in smooth mercuration of **I**–**IV** with replacement of the methylene hydrogen atom by the $HgOCOCF_3$ group. The reactions afford mercury-containing triphenylphosphonium salts **V**–**VIII** in quantitative yields (Table 1) and ethyl trifluoroacetate in 70–80% yield (GLC data).



$$[\mathrm{Ph_3\overset{+}{P}CH_2C(O)Ar}]X^- + \mathrm{Hg(OCOCF_3)_2}$$

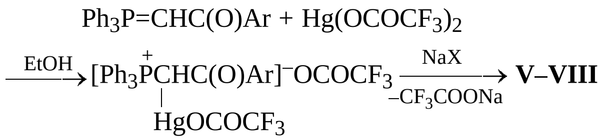
I–IV

$$\xrightarrow[-\mathrm{CF_3COOEt},\ -\mathrm{H_2O}]{\mathrm{EtOH}} [\mathrm{Ph_3\overset{+}{P}\underset{\mathrm{HgOCOCF_3}}{C}HC(O)Ar}]X^-,$$

V–VIII

Ar = Ph (**I, V**), 4-BrC_6H_4 (**II, IV, VI, VIII**), 4-$NO_2C_6H_4$ (**III, VII**); X = Br (**I–III, V–VII**), ClO_4 (**IV, VIII**).

The same salts were obtained (after treatment of the reaction mixture with NaBr or $NaClO_4$) by addition of mercury(II) trifluoroacetate to the corresponding aroyltriphenylphosphorylides [12] at room temperature in ethanol.



$$\mathrm{Ph_3P{=}CHC(O)Ar} + \mathrm{Hg(OCOCF_3)_2}$$

$$\xrightarrow{\mathrm{EtOH}} [\mathrm{Ph_3\overset{+}{P}\underset{\mathrm{HgOCOCF_3}}{C}HC(O)Ar}]^-\mathrm{OCOCF_3} \xrightarrow[-\mathrm{CF_3COONa}]{\mathrm{NaX}} \mathbf{V\text{–}VIII}$$

This fact suggests that mercuration of **I**–**IV** on heating in ethanol occurs via initial formation of phosphorylides, followed by addition of mercury(II) trifluoroacetate. The first step is apparently limiting, and the second step is fast.

Salts **V**–**VIII** are colorless or weakly colored solids melting with decomposition; they are readily soluble in polar organic solvents, poorly soluble in water, and insoluble in hydrocarbons and diethyl ether. Their composition and structure are confirmed by elemental analysis (Table 1), ^1H NMR and IR spectra (Table 2), and chemical transformations.

The presence of the covalent C–Hg bond in **V**–**VIII** is proved by coupling between the methine proton and ^{199}Hg nucleus, observed in the ^1H NMR spectrum, and also by a downfield shift of the methine proton signal due to magnetic anisotropy produced by the mercury-containing substituent [13].

The chemical properties of **V**–**VIII** are consistent with their structure. On heating in HCl, they decompose to give the starting salts **I**–**IV** and Hg^{2+} ions, which are quantitatively precipitated in the form of

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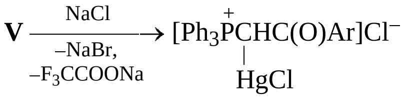
Table 1. Reaction times, yields, constants, and analytical data for **V–VIII, X–XV**, and **XVII–XXVII**

Comp. no.	Reaction time, min	Yield, %	Decomposition point, °C	Found, %		Formula	Calculated, %	
				Hg	N (Br)		Hg	N (Br)
V	60	98	105–107	25.74	(10.28)	$C_{28}H_{21}BrF_3HgO_3P$	25.93	(10.34)
VI	60	99	108–110	23.41	(18.53)	$C_{28}H_{20}Br_2F_3HgO_3P$	23.53	(18.77)
VII	60	99	100–102	23.59	1.90 (9.68)	$C_{28}H_{20}BrF_3HgNO_5P$	24.51	1.7 (9.77)
VIII	35	98	138–140	22.63	(13.08)[a]	$C_{28}H_{20}BrClF_3HgO_7P$	23.00	(13.25)[a]
X[b]	15	79	151–152	29.08	–	$C_{28}H_{20}F_3HgO_3P$	28.96	–
XI	10	77	149–150	26.82	(10.63)	$C_{28}H_{19}BrF_3HgO_3P$	26.00	(10.37)
XII	10	76	162–163	27.56	1.78	$C_{28}H_{19}F_3HgNO_5P$	27.20	1.90
XIII[c]	30	68	164–165	19.93	–	$C_{52}H_{40}HgO_2P_2$	20.93	–
XIV	30	65	168–170	18.00	(14.51)	$C_{52}H_{38}Br_2HgO_2P_2$	17.97	(14.33)
XV	10	90	135–136	18.73	3.00	$C_{52}H_{38}HgN_2O_6P_2$	19.71	2.75
XVII	60	73	139–140	33.08	(13.22)	$C_{17}H_{10}BrF_3HgO_3$	33.46	(13.34)
XVIII	60	71	165–166	30.85	2.31 (12.36)	$C_{17}H_9BrF_3HgNO_5$	31.12	2.17 (12.41)
XIX	90	70	184–186	32.18	(12.75)	$C_{17}H_{10}BrF_3HgO_4$	32.59	(13.00)
XX	90	30	175–176	31.64	(12.23)	$C_{19}H_{12}BrF_3HgO_4$	31.27	(12.47)
XXI	80	50	181–182	31.96	(12.64)	$C_{19}H_{11}BrF_3HgO_3$	32.12	(12.81)
XXII	120	58	173–174	35.28	2.36	$C_{17}H_{10}F_3HgNO_5$	35.47	2.48
XXIII	60	71	166–168	32.73	4.42	$C_{17}H_9F_3HgN_2O_7$	32.85	4.59
XXIV	120	46	193–195	34.31	2.43	$C_{17}H_{10}F_3HgNO_6$	34.49	2.41
XXV	100	59	185–188	32.83	2.45	$C_{19}H_{12}F_3HgNO_6$	33.02	2.30
XXVI	100	64	192–194	33.84	2.41	$C_{19}H_{11}F_3HgNO_5$	33.97	2.37
XXVII	30	56	201–203	23.35	3.18 (18.26)	$C_{30}H_{18}Br_2HgN_2O_6$	23.26	3.25 (18.55)

[a] Br + Cl. [b] Found P, %: 13.08. Calculated P, %: 13.25. [c] Found P, %: 6.54. Calculated P, %: 6.47.

HgS on passing H_2S through the reaction mixture after decomposition. On treatment of salt **V** with NaCl in aqueous alcohol on heating, it quantitatively transformed into known [4] chloride **IX** identified by the melting point and IR spectrum.

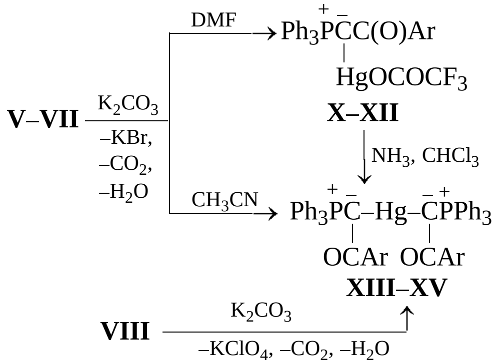


$$\mathbf{V} \xrightarrow[-\mathrm{NaBr},\ -\mathrm{F_3CCOONa}]{\mathrm{NaCl}} [\mathrm{Ph_3}\overset{+}{\mathrm{P}}\underset{\mathrm{HgCl}}{\mathrm{CH}}\mathrm{C(O)Ar}]\mathrm{Cl}^-$$

IX

We believe that formation of mercurated phosphorylides **X**–**XV** on treatment of **V**–**VIII** with aqueous sodium carbonate is a convincing chemical evidence of the covalent nature of the C–Hg bond in these compounds. The structure of the ylides formed significantly depended on the solvent in which the reaction was performed and also on the anion X^- (e.g., in **VI** and **VIII**). If the reaction was performed in dimethylformamide (DMF), bromides **V**–**VII** transformed into ylides **X**–**XII**, whereas in acetonitrile only symmetrical ylides **XIII**–**XV** were obtained. Treatment of perchlorate **VIII** with aqueous K_2CO_3 gave symmetrical ylide **XIV** only, irrespective of the solvent. We obtained the same symmetrical mercury-containing ylides **XIII**–**XV** by reactions of **X**–**XII** with ammonia in chloroform. Ammonia is known [14] to be a versatile symmetrizing agent for many organomercury salts.

It should also be noted that treatment of **IX** with potassium carbonate in DMF yields the known [3, 4] phosphorylide **XVI**, whereas in acetonitrile the symmetrical ylide **XIII** is formed, i.e., the pattern was similar to that noted above.



Ar = Ph (**X, XIII**), 4-BrC_6H_4 (**XI, XIV**), 4-$NO_2C_6H_4$ (**XII, XV**).

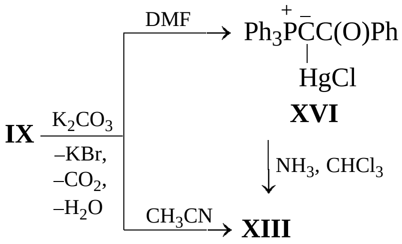
Table 2. IR and ^{1}H NMR data for **V–VIII, X–XV**, and **XVII–XXVII**

Comp. no.	IR spectrum, ν, cm^{-1}					^{1}H NMR spectrum, δ, ppm		
	aromatic system	C–O	C=O	C–Br (NO_2)	C=C	CH ($^{2(3)}J_{HgH}$, Hz)	$P(C_6H_5)_3$, m or R	Ar–CO
V	1602, 1587, 1501, 1473	1221	1672, 1620			6.62 m (272)	8.91–8.05	7.62–7.88 m (5H)
VI	1598, 1582, 1496, 1468	1218	1678, 1622	760		6.66 m (275)	8.92–8.08	7.72–7.96 m (4H)
VII	1600, 1589, 1506, 1463	1220	1680, 1638	(1352)		6.71 m (283)	8.96–8.12	7.83–8.05 d.d (4H)
VIII	1598, 1583, 1496, 1463	1218	1679, 1622	762		6.65 m (276)	8.94–8.10	7.73–7.98 m (4H)
X	1605, 1578, 1508, 1463	1231	1660, 1562				8.78–8.02	7.50–7.72 m (5H)
XI	1603, 1581, 1503, 1462	1228	1662, 1560	760			8.80–8.02	7.68–7.85 m (4H)
XII	1606, 1587, 1510, 1471	1225	1668, 1572	(1350)			8.90–8.04	7.65–7.92 d.d (4H)
XIII	1607, 1592, 1508, 1463		1568				8.62–7.97	7.42–7.63 m (5H)
XIV	1605, 1582, 1504, 1471		1562	761			8.60–7.93	7.58–7.82 m (4H)
XV	1608, 1591, 1512, 1475		1570	(1348)			8.72–8.01	7.58–7.84 d.d (4H)
XVII	1545, 1500, 1440	1218	1672, 1625	762	1600	6.93 s (598)	8.36–8.03 m (5H)	8.92–8.76 d.d (4H)
XVIII	1546, 1498, 1445	1220	1675, 1627	760, (1356)	1598	6.89 s (588)	8.38–8.12 d.d (4H)	8.94–8.78 d.d (4H)
XIX	1562, 1502, 1458	1222, 3560[a]	1677, 1625	762	1602	6.90 s (602)	8.25–7.93 d.d (4H)	8.91–8.69 d.d (4H)
XX	1583, 1532, 1480, 1463	1256, 1217	1678, 1632	760	1598, 1618	6.98 m (628)	6.76 m (2H), 7.58 s (1H), 6.10–5.87 m (4H)	8.90–8.63 d.d (4H)
XXI	1546, 1502, 1448	1228	1675, 1628	761	1600, 1615	6.91 m (625)	7.68–7.50 m (5H), 6.53–6.45 m (2H)	8.91–8.68 d.d (4H)
XXII	1558, 1499, 1462	1222	1682, 1631	(1352)	1601	6.98 s (628)	8.52–8.11 m (5H)	9.36–8.93 d.d (4H)
XXIII	1561, 1503, 1468	1225	1675, 1638	(1350)	1596	7.02 s (634)	9.02–8.90 d.d (4H)	9.43–9.13 d.d (4H)
XXIV	1565, 1504, 1460	1225, 3562[a]	1680, 1628	(1352)	1602	6.99 s (629)	8.31–7.98 d.d (4H)	9.41–9.08 d.d (4H)
XXV	1587, 1541, 1477, 1458	1254, 1218	1678, 1630	(1350)	1600, 1617	6.91 m (629)	6.77 m (2H), 7.62 s (1H), 6.15–5.94 m (4H)	9.39–9.06 d.d (4H)
XXVI	1545, 1501, 1452	1226	1678, 1626	(1352)	1601, 1617	6.95 m (628)	7.75–7.53 m (5H), 6.64–6.54 m (2H)	9.37–8.98 d.d (4H)
XXVII	1583, 1551, 1493, 1460		1628	762, (1350)	1602	6.82 s (486)	8.26–8.01 d.d (8H)	8.72–8.38 d.d (8H)

[a] ν_{OH}.

The significant effects of solvent and anion X^- in salts **VI** and **VIII** on the composition of the reaction products may be due to many factors, among which the most significant, in our opinion, are the solvation effects and the solubilitites of the corresponding potassium salts formed as by-products.

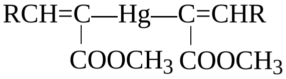
Mercury-containing phosphorylides **X**–**XV** are

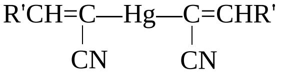


colorless or pale yellow substances readily soluble in polar organic solvents and insoluble in water. The analytical (Table 1), ^1H NMR, and IR data (Table 2) are consistent with their composition and structure. In the IR spectra, the absorption band of the carbonyl group in the aroyl moiety is strongly shifted toward lower frequencies (1570–1560 cm^{-1}), which is typical of related ylides [4, 8]. In the ^1H NMR spectra, the methine proton signal is absent, and the aromatic proton signals are shifted upfield by 0.1–0.2 ppm owing to decreased positive charge on the P atom.

Upon refluxing in 15% HCl, all the ylides **X**–**XV** eliminated mercury in the form of Hg^{2+}, which was quantitatively precipitated on passing H_2S through the reaction mixture.

It is known [10] that triphenylphosphonium ylides are widely used in the Wittig reaction [15] with carbonyl compounds to prepare the corresponding olefins. Mercury-containing symmetrical ylides **XIII**–**XV** were also used [6] in this reaction with a series of aromatic aldehydes and trichloroacetaldehyde; the following unsaturated mercury-containing compounds were obtained in 43–71% yields:

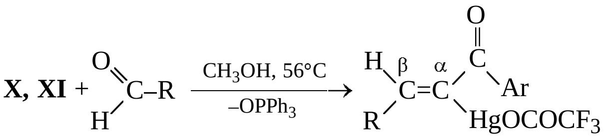

$RCH{=}\underset{COOCH_3}{C}{—}Hg{—}\underset{COOCH_3}{C}{=}CHR$


$R'CH{=}\underset{CN}{C}{—}Hg{—}\underset{CN}{C}{=}CHR'$

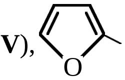
R = Ph, 4-$MeOC_6H_4$, 4-BrC_6H_4, 3-$NO_2C_6H_4$, 4-$NO_2C_6H_4$;
R' = 4-$NO_2C_6H_4$, CCl_3.

In this work, we studied new examples of the Wittig reaction between unsymmetrical ylides **X** and **XI** and a series of aromatic aldehydes, including those containing double bonds. We found the best reaction conditions: refluxing of equimolar amounts of the reactants in a minimal volume of methanol for 1–2 h. The resulting unsaturated organomercury compounds precipitated on cooling, and triphenylphosphine oxide remained in the solution. The yields of products **XVII**–**XXVI** were 30–72% (Table 1).

The compositions and structures of olefins **XVII**–**XXVI** were confirmed by elemental analysis (Table 1), ^1H NMR and IR spectra (Table 2), and chemical transformations. The IR spectra contained absorption bands in the range 1620–1640 cm^{-1} characterizing the stretching vibrations of the C=O bond in the aroyl moiety conjugated with the double bond, and also the bands of the $HgOCOCF_3$ group. In addition, the IR spectra contained the absorption bands of the other functional groups present in the products (NO_2, OH, double bonds, aromatic system).



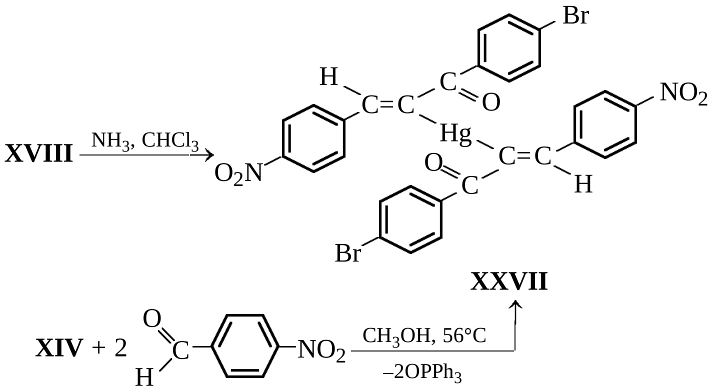
XVII–XXVI

A = 4-BrC_6H_4 (**XVII–XXI**), 4-$NO_2C_6H_4$ (**XXII–XXVI**);
R = Ph (**XXVII, XXII**), 4-$NO_2C_6H_4$ (**XVIII, XXIII**);
4-HOC_6H_4 (**XIX, XXIV**), CH=CH–CH=CH– (**XX, XXV**), PhCH=CH– (**XXI, XXVI**).

The ^1H NMR spectra (Table 2) of **XVII**–**XXVI** contain three groups of proton signals. The aryl protons are manifested in the low field as a doublet of doublets with characteristic proton coupling constants; the protons of radical R are manifested in the higher field, also as a doublet of doublets for compounds with *p*-substituents in the benzene ring (**XVIII**, **XIX**, **XXIII**, **XXIV**) and as a multiplet for **XVII**, **XX**–**XXII, XXV**, and **XXVI**; olefin protons give complex multiplets in the range 5.87–6.53 ppm. The $C^{\beta}H$ signal is observed at 6.89–7.02 ppm and is characterized by the coupling constant $^3J_{HgH}$ as large as 634–586 Hz. Such values of $^3J_{HgH}$ are typical [17, 18] of the transoid arrangement of the H and Hg atoms at the C=C bond. Hence, compounds **XVII**–**XXVI** can be identified as *Z* isomers.

An additional evidence of the *Z* structure of the olefins prepared is the symmetrization reaction (see below the example for **XVIII**) occurring under the action of NH_3 in $CHCl_3$ at room temperature. The reaction yielded a symmetrical mercury-containing compound **XXVII**; in its ^1H NMR spectrum, the olefin proton signals were observed in a higher field, but with approximately the same coupling constant $^3J_{HgH}$ (Table 2) characteristic of *Z* isomers. We also prepared **XXVII** by an independent synthesis: reaction of symmetrical ylide **XIV** with *p*-nitrobenzaldehyde in methanol. It is known [6] that such reactions yield exclusively the *Z* isomers, and this was the case in our reaction.

Samples of compound **XXVII** prepared by different routes appeared to be identical in physicochemical properties and in the ^1H NMR and IR spectra.



EXPERIMENTAL

The IR spectra were recorded on IKS-29 and UR-20 spectrophotometers (mulls in mineral oil or KBr pellets). The ^{1}H NMR spectra were taken on a Bruker WP-200SY spectrometer (200.13 MHz, DMSO-d_6, internal reference TMS).

[Benzoyl(trifluoroacetatomercurio)methyl]triphenylphosphonium bromide V. *a*. A 1-mmol portion of mercury(II) trifluoroacetate was added to a solution of 1 mmol of phenacyltriphenylphosphonium bromide **I** [11] in 10 ml of anhydrous ethanol. The mixture was refluxed for 1 h. After cooling, 30 ml of diethyl ether was added, and the mixture was allowed to stand at 5–6°C for 15 h. The precipitate was filtered off, washed with 10 ml of hexane, and dried. Yield of salt **V** 0.76 g. Compounds **VI**–**VIII** were prepared similarly.

b. To a solution of 1 mmol of benzoyltriphenylphosphorylide [12] in 5 ml of ethanol, we added 1 mmol of mercury(II) trifluoroacetate and 0.2 g of NaBr. The mixture was stirred for 30 min at room temperature, after which 30 ml of diethyl ether was added, and the mixture was allowed to stand at 5–6°C for 15 h. The precipitate was filtered off, washed with 5 ml of cold water, and dried. Yield of salt **V** 0.75 g. Compounds **VI**–**VIII** were prepared similarly.

[Benzoyl(chloromercurio)methyl]triphenylphosphonium chloride IX. To a solution of 0.5 mmol of salt **V** in 3 ml of ethanol, we added 3 ml of 20% aqueous NaCl; the mixture was refluxed for 1 h. After cooling, 5 ml of water was added, and the mixture was allowed to stand at 5–6°C for 15 h. The precipitate was filtered off, washed with 5 ml of cold water, and dried. Yield of salt **IX** 0.32 g, mp 188–189°C [3].

Benzoyl(trifluoroacetatomercurio)triphenylphosphoniomethylide X. To a solution of 0.5 mmol of **V** in 5 ml of DMF, we added 0.8 ml of a 10% aqueous solution of potassium carbonate. The mixture was stirred for 15 min at room temperature. Then 10 ml of water was added, and the mixture was allowed to stand at 5–6°C for 15 h. The precipitate was filtered off, washed with 5 ml of water, and dried. Yield of ylide **X** 0.30 g. Ylides **XI** and **XII** were prepared similarly.

Bis[benzoyl(triphenylphosphonio)methylido]mercury XIII. *a*. To a solution of 0.5 mmol of **V** in 5 ml of acetonitrile, we added 0.8 ml of a 10% aqueous solution of potassium carbonate. The mixture was stirred for 30 min at room temperature. Then 10 ml of water was added, and the mixture was allowed to stand at 5–6°C for 15 h. The precipitate was filtered off, washed with 5 ml of water, and dried. Yield of diylide **XIII** 0.16 g. Compounds **XIV** and **XV** were prepared similarly.

b. To 0.5 mmol of ylide **X**, we added 5 ml of a saturated (at room temperature) solution of ammonia in chloroform. The mixture was stirred for 30 min. Then the solvent was removed in a vacuum at room temperature, and the residue was washed with 10 ml of water and dried. Yield of diylide **XIII** 0.12 g. Diylides **XIII**–**XV** were prepared similarly from ylides **XI**, **XII**, and **XVI**.

(*Z*)-1-(4-Bromophenyl)-2-trifluoroacetatomercurio-3-phenyl-2-propen-1-one XVII. To a solution of 1 mmol of ylide **XI** in 10 ml of methanol, we added 1 mmol of benzaldehyde; the mixture was refluxed for 1 h. The precipitate that formed after cooling was filtered off, washed with 3 ml of cold methanol, and dried. Yield of **XVII** 0.44 g. Triphenylphosphine oxide was isolated from the filtrate after methanol evaporation; yield 82%, mp 155–156°C (from aqueous ethanol).

Compounds **XVIII**–**XXVI** were prepared similarly.

Bis[2-(4-nitrophenyl)-1-(4-bromobenzoyl)ethenyl]mercury XXVII. *a*. To 0.5 mmol of **XVIII**, we added 3 ml of a saturated (at room temperature) solution of ammonia in chloroform. The mixture was stirred for 30 min. The solvent was removed in a vacuum at room temperature, and the residue was washed with 8 ml of water and dried. Yield of **XXVII** 0.12 g.

b. To a solution of 0.5 mmol of diylide **XIV** in 6 ml of methanol, we added 1 mmol of 4-nitrobenzaldehyde; the mixture was refluxed for 1 h. The precipitate that formed after cooling was filtered off, washed with 3 ml of cold methanol, and dried. Yield of **XXVII** 0.17 g. Triphenylphosphine oxide was isolated from the filtrate after methanol evaporation; yield 79%, mp 156–157°C (from aqueous ethanol).

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