

## Organometallic Chemistry

### R'Hg- and Ph<sub>3</sub>PAu-Derivatives of substituted malononitriles RCH(CN)<sub>2</sub>

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The organomercurials RC(HgR')(CN)<sub>2</sub> (R = Me, Ph; R' = Ph, CH<sub>2</sub>Ph) have been obtained by the action of organomercury hydroxides R'HgOH or acetates R'HgOAc on substituted malonodinitriles RCH(CN)<sub>2</sub>. (Ph<sub>3</sub>PAu)<sub>3</sub>O<sup>+</sup>BF<sub>4</sub><sup>-</sup> reacts with the same nitriles to give organogold derivatives RC(AuPPh<sub>3</sub>)(CN)<sub>2</sub>. The study of the structures of organomercury and -gold compounds by <sup>1</sup>H, <sup>31</sup>P and <sup>199</sup>Hg NMR spectra as well as by IR spectra show that these compounds exist mainly in the C-form. The degenerated exchange reaction involving the cleavage of C—Hg bond takes place in the phenylmercury derivative of methylmalonodinitrile. Organomercury derivatives of substituted malonodinitriles are stable with respect to symmetrization in solution, in contrast to PhCH(HgPh)CN studied previously.

**Key words:** methyl- and phenylmalononitriles; RHg- and Ph<sub>3</sub>PAu-derivatives; <sup>1</sup>H, <sup>31</sup>P, <sup>199</sup>Hg NMR spectra; IR spectra; C—Hg bond; exchange reactions.

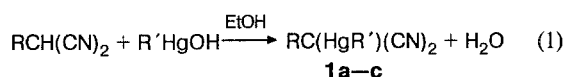
Unsymmetrical organomercury derivatives of  $\alpha,\alpha$ -dinitriles RC(HgR')(CN)<sub>2</sub> (**1**) as well as derivatives of triphenylphosphinogold RC(Ph<sub>3</sub>PAu)(CN)<sub>2</sub> (**2**) are not described in literature. The diaurated derivative of malonodinitrile, bis(triphenylphosphinogold)dicyanomethane (Ph<sub>3</sub>PAu)<sub>2</sub>C(CN)<sub>2</sub> (**3**), formed in the reaction of malonodinitrile with tris(triphenylphosphinogold)oxonium tetrafluoroborate in the presence of bases,<sup>1</sup> has been obtained and characterized by X-ray analysis.

Organometallic derivatives of the R'C(ML<sub>n</sub>)(CN)<sub>2</sub> type are convenient model compounds for studying dynamic processes with cleavage of the L<sub>n</sub>M—C bond.

Methylmalonodinitrile MeCH(CN)<sub>2</sub> (**4**) and phenylmalonodinitrile PhCH(CN)<sub>2</sub> (**5**) were chosen as the

initial dinitriles. The selection of the mentioned compounds was based on the fact that data on CH-acidity in DMSO for them and CH<sub>2</sub>(CN)<sub>2</sub> are presented in literature.<sup>2</sup> This fact is of great importance for the quantitative determination of the relative polarity of the element—hydrogen and element—metal bonds in HX-acids and their organometallic derivatives by the exchange equilibria method (see, for example, Ref. 3 and references cited therein).

We have established that the interaction of organomercury hydroxides R'HgOH (R' = Ph, PhCH<sub>2</sub>) with **5** or PhHgOH with **4** in ethanol results in mercuration of the considered dinitriles, and compounds **1a—c** are formed in quantitative yields.



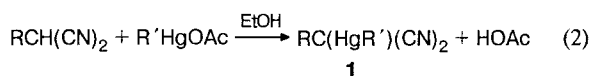
a: R = Me, R' = Ph

b: R = R' = Ph

c: R = Ph, R' = PhCH<sub>2</sub>

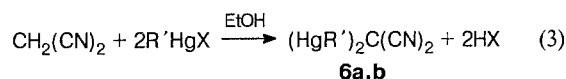
The reaction of dinitrile **4** with PhCH<sub>2</sub>HgOH is accompanied by the liberation of metallic mercury.

Mercuration of dinitriles **4** and **5** can also be performed by organomercury acetates R'HgOAc in the presence of catalytic amounts (~5 %) of a base (KOH) or even without a catalyst.



This method of synthesis of compounds **1** is simpler because it does not require preliminary preparation of hydroxides R'HgOH, which are synthesized by alkaline hydrolysis of acetates R'HgOAc.<sup>4</sup> However, the yields and purity of the corresponding products of reaction (2) are considerably lower than those in reaction (1). In particular, PhCH<sub>2</sub>HgOAc does not mercurate dinitrile **4** under the same conditions.

We also found that R'HgOH and R'HgOAc mercurate unsubstituted malonodinitrile CH<sub>2</sub>(CN)<sub>2</sub> with substitution of both acidic CH-protons, resulting in the formation of dimercurated compounds **6a,b**.

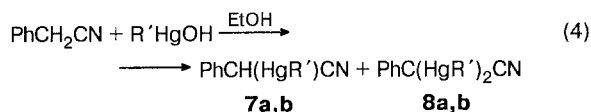


X = OH, OAc

a: R' = Ph

b: R' = PhCH<sub>2</sub>

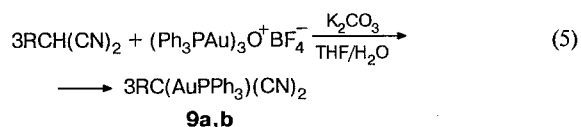
It should be noted that mercuration of a less acidic mononitrile, PhCH<sub>2</sub>CN, leads to the formation of a mixture of mono- and dimercurated products, as we showed previously.<sup>5</sup>



a: R' = Me

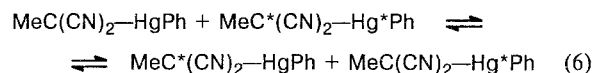
b: R' = Ph

Dinitriles **4** and **5** are auriated<sup>1</sup> with tris(triphenylphosphinogold)oxonium tetrafluoroborate to form compounds **9a,b**.

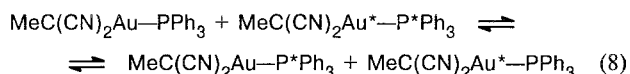
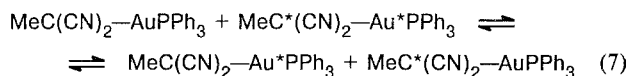


The structures of the obtained compounds in solution were studied by <sup>1</sup>H, <sup>31</sup>P and <sup>199</sup>Hg NMR spectroscopy as well as IR spectroscopy (Table 1).

The <sup>1</sup>H NMR spectrum of the solution of dinitrile **1a** in C<sub>5</sub>D<sub>5</sub>N at ~20 °C contains one broadened Me-group signal (ν<sub>1/2</sub> = 9 Hz). At -50 °C this signal becomes narrow with narrow satellites caused by the spin-spin interaction of the <sup>199</sup>Hg nucleus with protons of the methyl group, the value of <sup>3</sup>J<sub>H,199Hg</sub> is 86 Hz. A similar situation with satellites of mercury, but already at ~20 °C, is observed for a solution of compound **1a** in a C<sub>5</sub>D<sub>5</sub>N-CDCl<sub>3</sub> mixture\* (<sup>3</sup>J<sub>H,199Hg</sub> = 81 Hz) as well as in CD<sub>2</sub>Cl<sub>2</sub> (<sup>3</sup>J<sub>H,199Hg</sub> = 87 Hz). The data obtained indicate the existence of degenerate intermolecular exchange of PhHg-fragments in a solution of **1a** in C<sub>5</sub>D<sub>5</sub>N.



The <sup>1</sup>H NMR spectrum of the gold-containing compound **9a** in CDCl<sub>3</sub> contains a doublet signal of Me-group protons with <sup>4</sup>J<sub>H,31P</sub> = 3.9 Hz. In the <sup>1</sup>H NMR spectrum of the solution of the same compound in C<sub>5</sub>D<sub>5</sub>N, the Me-group protons manifest themselves as a narrow singlet. This fact may attest to lability in the H-C-C-Au-P fragment either of the C-Au bond, which corresponds to the degenerate exchange of Ph<sub>3</sub>PAu-fragments (reaction (7)), or of the Au-P bond, which corresponds to the intermolecular exchange of phosphine ligands (reaction (8)).



It should be mentioned that the exchange reaction of triphenylphosphine ligands in the mixture of **9a** and **9b** in pyridine is fast in the time scale of <sup>31</sup>P NMR, because the <sup>31</sup>P NMR spectrum of the solution of this mixture contains one broadened signal of phosphorus, whose chemical shift (35.2 ppm) is intermediate between the chemical shifts of the signals of the individual compounds (38.1 and 31.0 ppm, respectively). At the same time, the <sup>31</sup>P NMR spectrum of a solution of a similar mixture in CHCl<sub>3</sub> contains two signals, whose positions correspond to the signals of the individual compounds (38.0 and 36.8 ppm, respectively).

We intend to perform a comprehensive study of the exchange kinetics (see reaction (6)) in the future. Let us also indicate that the C-H bond in the initial dinitrile **4** is stable in the time scale of NMR in CDCl<sub>3</sub> and C<sub>5</sub>D<sub>5</sub>N under normal conditions. The existence of a

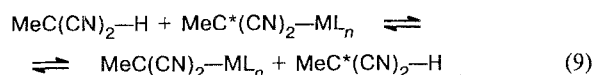
\* Compound **1a** is insoluble in pure CDCl<sub>3</sub>.

**Table 1.** NMR and IR spectroscopy data on mercury- and gold-containing derivatives of malononitrile and its substituted derivatives

Compound	Solvent	NMR spectra		IR spectrum, $\nu(\text{C}\equiv\text{N})/\text{cm}^{-1}$ (hexachlorobutadiene)
		$\delta$ $^1\text{H}$	$\delta$ $^{199}\text{Hg}$ (pyridine)	
<b>MeC(HgPh)(CN)<sub>2</sub></b> <b>(1a)</b>	$\text{C}_5\text{D}_5\text{N}$	1.974 ( $\text{CH}_3$ ); 6.99–7.78 (m, Ph)	–1137.6	2223 s, 2237 s, 2250 sh
	$\text{C}_5\text{D}_5\text{N}-\text{CDCl}_3$ (1 : 1)	1.890 ( $^3J_{\text{H},^{199}\text{Hg}} = 81$ Hz, $\text{CH}_3$ ); 7.28–7.72 (m, Ph)		
	$\text{CD}_2\text{Cl}_2$	2.028 ( $^3J_{\text{H},^{199}\text{Hg}} = 87$ Hz, $\text{CH}_3$ ); 7.11–7.53 (m, Ph)		
	$(\text{CD}_3)_2\text{SO}$	1.890 ( $\text{CH}_3$ ); 7.06–7.81 (m, Ph)		
<b>PhC(HgPh)(CN)<sub>2</sub></b> <b>(1b)</b>	$\text{C}_5\text{D}_5\text{N}$	6.77–8.10 (m, Ph)	–1291.5	2242 s
<b>PhC(HgCH<sub>2</sub>Ph)(CN)<sub>2</sub></b> <b>(1c)</b>	$\text{C}_5\text{D}_5\text{N}$	3.344 ( $^2J_{\text{H},^{199}\text{Hg}} = 301$ Hz, $\text{CH}_3$ ); 6.77–7.57 (m, Ph)	–1253.4	2232 s
<b>C(HgPh)<sub>2</sub>(CN)<sub>2</sub></b> <b>(6a)</b>	$\text{C}_5\text{D}_5\text{N}$	7.00–7.71 (m, Ph)	–924.0	2210 s, 2251 w
<b>C(HgCH<sub>2</sub>Ph)<sub>2</sub>(CN)<sub>2</sub></b> <b>(6b)</b>	$\text{C}_5\text{D}_5\text{N}$	2.687 ( $^2J_{\text{H},^{199}\text{Hg}} = 214$ Hz, $\text{CH}_3$ ); 6.85–7.19 (m, Ph)		2180 w
<b>MeC(AuPPh<sub>3</sub>)(CN)<sub>2</sub></b> <b>(9a)</b>	$\text{C}_5\text{D}_5\text{N}$	2.006 ( $\text{CH}_3$ ); 7.37–7.57 (m, Ph)		2197 m, 2211 s
	$\text{CDCl}_3$	1.939 d ( $^4J_{\text{H},^{31}\text{P}} = 3.9$ Hz, $\text{CH}_3$ ); 7.42–7.55 (m, Ph)		
<b>PhC(AuPPh<sub>3</sub>)(CN)<sub>2</sub></b> <b>(9b)</b>	$\text{C}_5\text{D}_5\text{N}$	6.72–7.56 (m, Ph)		2216 s
<b>MeCH(CN)<sub>2</sub></b> <b>(4)</b>	$\text{C}_5\text{D}_5\text{N}$	1.634 (d, $^3J_{\text{H},\text{H}} = 7.2$ Hz, $\text{CH}_3$ ); 5.022 (q, CH)		2281 s
	$(\text{CD}_3)_2\text{SO}$	1.622 (d, $^3J_{\text{H},\text{H}} = 7.3$ Hz, $\text{CH}_3$ ); 5.010 (q, CH)		
<b>PhCH(CN)<sub>2</sub></b> <b>(5)</b>	$\text{CDCl}_3$	5.061 (CH); 7.498 (Ph)		2270 s

doublet of Me-group protons attests to this (see Table 1). This conforms to the literature data, according to which fast proton exchange in the C—H bonds of hydrocarbons at elevated acidity occurs only under catalytic conditions.<sup>6</sup>

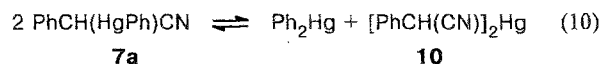
It is important for our purposes that in mixtures of dinitrile **4** and its organometallic derivatives, exchanges of the mercury—hydrogen and gold—hydrogen types in  $C_5D_5N$  (reaction (9)) are slow in the NMR time scale (the spectrum contains individual signals of Me-group protons).


$$ML_n = \text{HgR}, \text{AuPPh}_3$$

In the light of the known ability of  $\text{RHgX}$  compounds to undergo symmetrization,<sup>7,8</sup> the problem of the stability of the synthesized organomercury derivatives in solutions seems also important. This information can be obtained from the  $^{199}\text{Hg}$  NMR spectra of these compounds (see Table 1). The  $^{199}\text{Hg}$  NMR spectra of freshly prepared solutions of **1a–c** and **2a,b** in pyridine contains one signal of the mercury nucleus related to the unsymmetrical compound. The spectra of **1a–c** and **2b** do not change after they are allowed to stand for a week,

whereas in the spectrum of dimercurated compound **6a** two additional weak signals appear at  $-792$  and  $-1051$  ppm. The weak-field signal relates to  $\text{Ph}_2\text{Hg}$  and that of the high field seems to be attributed to another symmetric compound,  $\text{Hg}[\text{PhHgC}(\text{CN})_2]_2$ .

Thus, the organomercury derivatives of dinitriles **1a–c** considered as model compounds are stable with respect to symmetrization in solution in contrast to the derivative of mononitrile **7a**. As we established previously,<sup>5</sup> the latter compound is partially symmetrized in solution (in  $\text{CHCl}_3$  and pyridine).



$\delta$ $^{199}\text{Hg}$ ( $\text{C}_5\text{H}_5\text{N}$ )	-1043 ppm	-792 ppm	-1261 ppm
			-1263 ppm

Let us note that, in addition to the data presented in literature<sup>5</sup> on the <sup>199</sup>Hg NMR spectrum of compound **7a** in C<sub>5</sub>H<sub>5</sub>N, the existence of compound **10** in the system (see reaction (10)) is confirmed by the <sup>199</sup>Hg NMR spectrum of a previously known sample of **10**.<sup>\*</sup> The <sup>199</sup>Hg NMR spectrum of a pyridine solution of **10**

\* The authors are grateful to coworkers of the laboratory of N. S. Vyazankin for the synthesis of compound **10**.

contains two closely arranged signals of the mercury nuclei with  $-1261.8$  and  $-1263.3$  ppm of equal intensities. The doubling of the mercury signals seems to be connected with the existence of two asymmetric carbon atoms in compound **10** and is caused by the different screening of mercury in the meso-forms of **10a** and enantiomers of **10b**.

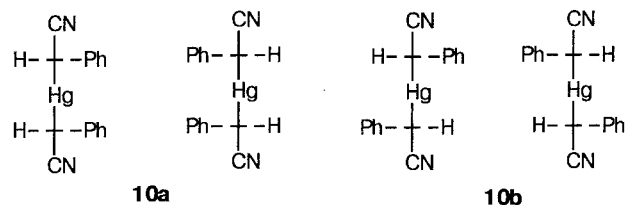


Table 1 also presents the frequencies of the valent oscillations of the  $\text{C}\equiv\text{N}$  group for solutions of the considered compounds in hexachlorobutadiene. These frequencies for metal-containing compounds are within the range  $2180$  to  $2220\text{ cm}^{-1}$ , i.e., they are essentially shifted to low frequencies as compared with the spectra of the initial dinitriles.

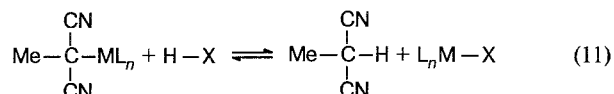
The data of NMR and IR spectroscopy attest that synthesized mercury- and gold-containing derivatives mainly exist in the C-form, like the monometallated (**7a,b**,  $^5\text{ MeHgCH}_2\text{CN}$  **10**) and dimetallated (**8a,b**  $^5$  and  $(\text{Ph}_3\text{PAu})_2\text{C}(\text{CN})_2$  **1**) C-derivatives of other  $\alpha$ -nitriles.

The data obtained allow one to make, in particular, the following conclusions.

1. Monometallated Hg- and Au-containing derivatives of dinitriles **1** and **8** in solutions are stable compounds and exist mainly in the C-form.

2. Screening of the Me-group in the initial nitrile **4** and its RHg- and  $\text{Ph}_3\text{PAu}$ -derivatives strongly differ.

3. Exchange of the Hg—H and Au—H type in the system (see reaction (9)) is slow in the  $^1\text{H}$  NMR time scale. All of these facts allow one to consider nitriles **4** and **5** and their PhHg- and  $\text{Ph}_3\text{Au}$ -derivatives as convenient model systems for the  $^1\text{H}$  NMR study of the position of mercury—hydrogen and gold—hydrogen type exchange equilibria in mixtures containing dinitrile **4**, other HX-acids, and their organometallic derivatives.



## Experimental

NMR spectra were recorded on a Bruker WP-200 SY spectrometer with operating frequencies of  $200.13\text{ MHz}$  ( $^1\text{H}$ ),  $81.01\text{ MHz}$  ( $^{31}\text{P}$ ), and  $35.76\text{ MHz}$  ( $^{199}\text{Hg}$ ). The values of  $\delta\text{ }^1\text{H}$  were determined relative to the residual hydrogen signal in  $\text{CDCl}_3$  ( $7.24$ ) and  $\text{C}_5\text{D}_5\text{N}$  ( $8.71$ ) and recalculated relative to TMS. The values of  $\delta\text{ }^{31}\text{P}$  were obtained by the exchange method relative to  $85\%\text{ H}_3\text{PO}_4$ , the accuracy of determination was  $\pm 0.5$ . The values of  $\delta\text{ }^{199}\text{Hg}$  were estimated by the exchange method relative to a  $0.2\text{ M}$  solution of  $\text{Ph}_2\text{Hg}$  in  $\text{C}_5\text{H}_5\text{N}$  and then recalculated with respect to  $\text{Me}_2\text{Hg}$  using the previously found<sup>10</sup> value of  $\delta\text{ }^{199}\text{Hg}$  for  $\text{Ph}_2\text{Hg}$  in  $\text{C}_5\text{H}_5\text{N}$  equal to  $-791.1$ . The accuracy of the determination of  $\delta\text{ }^{199}\text{Hg}$  was  $\pm 0.5$ . The stabilization of the resonance conditions in the case of  $^{31}\text{P}$  and  $^{199}\text{Hg}$  NMR spectra was performed by the signal of deuterium from  $\text{D}_2\text{O}$  placed between walls of the ampule ( $d = 10\text{ mm}$ ) and the shell with the sample ( $d = 8\text{ mm}$ ).

**Table 2.** Melting points and results of elemental analysis of compounds  $\text{L}_n\text{MCR}(\text{CN})_2$  and  $(\text{R}'\text{Hg})_2\text{C}(\text{CN})_2$  ( $\text{L}_n\text{M} = \text{R}'\text{Hg}$ ,  $\text{Ph}_3\text{PAu}$ )

Compound	Yield (%)	M.p./°C	Found Calculated (%)					Molecular formula
			C	H	N	M	P	
<b>1a</b>	80	177—179	33.62	2.00	7.83	55.75	—	$\text{C}_{10}\text{H}_8\text{HgN}_2$
			33.61	2.24	7.84	56.30	—	
<b>1b</b>	70	125—127	42.19	2.39	6.46	46.35	—	$\text{C}_{15}\text{H}_{10}\text{HgN}_2$
			42.95	2.38	6.69	47.97	—	
<b>1c</b>	60	93—95	44.08	2.78	6.85	45.98	—	$\text{C}_{16}\text{H}_{12}\text{HgN}_2$
			44.38	2.77	6.47	46.37	—	
<b>6a</b>	96	270—272	29.34	1.84	—	63.21	—	$\text{C}_{15}\text{H}_{10}\text{Hg}_2\text{N}_2$
			29.23	1.64	—	64.61	—	
<b>6b</b>	90	165—170	31.51	2.20	4.19	60.10	—	$\text{C}_{17}\text{H}_{14}\text{Hg}_2\text{N}_2$
			31.52	2.16	4.32	61.99	—	
<b>9a</b>	61	168—170	49.04	3.36	5.02	—	5.86	$\text{C}_{22}\text{H}_{18}\text{AuN}_2\text{P}$
			49.08	3.11	5.02	—	5.75	
<b>9b</b>	72	124—125	53.70	3.43	4.47	—	5.21	$\text{C}_{27}\text{H}_{20}\text{AuN}_2\text{P}$
			53.97	3.35	4.66	—	5.15	

Solvents were purified by standard procedures. Dinitrile of methylmalonic acid (**4**) and dinitrile of phenylmalonic acid (**5**) were prepared as described previously.<sup>12,13</sup>

**Synthesis of 1,1-dicyanoethylphenylmercury (1a).** A solution of 1.2 g (4 mmol) of PhHgOH in 30 mL of EtOH was added to a solution of 0.3 g (4 mmol) of dinitrile of methylmalonic acid in 15 mL of EtOH. The reaction mixture was stirred for 1.5 h at ~20 °C and allowed to stand for a night in a refrigerator. Precipitated colorless needle-shaped crystals of **1a** dried over alkali had a m.p. of 177–179 °C. Additional white crystals of **1a** (with the same m.p.) were obtained by evaporation of the mother liquor. The yield was 1.2 g (80 %).

The compounds **1b–d**, **6a,b** were obtained similarly.

**Synthesis of (α,α-dicyanobenzyl)benzylmercury (6b).** A solution of 0.84 g (2.5 mmol) of benzylmercury acetate in 30 mL of EtOH was added to a solution of 0.36 g (2.5 mmol) of dinitrile of phenylmalonic acid in 15 mL of EtOH. The reaction mixture was stirred for 1.5 h at ~20 °C, the solvent was removed under reduced pressure, and the residue was recrystallized from ethanol. Light-yellow crystals of **6b** (0.65 g, 60 %) were obtained, m.p. was 93–95 °C.

**Synthesis of 1,1-dicyanoethyl- and α,α-dicyanobenzyl-triphenylphosphinogold (9a,b).** A mixture of 0.082 g (1.05 mmol) of dinitrile of methylmalonic acid, 0.05 g of K<sub>2</sub>CO<sub>3</sub> and 0.3 mL of H<sub>2</sub>O in 30 mL of anhydrous THF was stirred for 20 min, then 0.5 g (0.34 mmol) of tris(triphenylphosphinogold)oxonium tetrafluoroborate was added. The mixture was stirred for 1.5 h more until the full dissolution of oxonium salt, the solvent was removed under reduced pressure (in the case when oil remained after the removal of the solvent, 3 mL of THF was added to the oil, and the solution was added dropwise with intense stirring to 50 mL of an ether–hexane (1 : 1) mixture, the precipitated powder was removed and dissolved in acetone). The residue was dissolved in a minimum amount of acetone, and 0.32 g (61 %) of compound **9a** was precipitated with an ether–hexane (1 : 2) mixture as a colorless crystalline substance with m.p. equal to 168–170 °C. Similarly, 0.44 g (72 %) of product **9b** as colorless crystals with m.p. 124–125 °C was obtained from 0.14 g (1.05 mmol) of dinitrile of phenylmalonic acid and 0.5 g (0.34 mmol) of the oxonium salt.

The data of elemental analysis and melting points of the compounds obtained are presented in Table 2.

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