# Gas-liquid chromatography of perfluorocarboxylic thioesters
## 2.* Sorption characteristics

*O. A. Osipova, V. E. Kirichenko, and K. I. Pashkevich**

*Institute of Organic Synthesis, Ural Branch of the Russian Academy of Sciences,*
*20 ul. S. Kovalevskoi, 620219 Ekaterinburg, Russian Federation.*
*Fax: 007 (343 2) 44 0458*

Perfluorocarboxylic thioesters $R_FC(O)SR$ ($R_F = CF_3(CF_2)_j$, $R = CH_3(CH_2)_i$, $i$ and $j = 0-5$) were studied for the first time by GLC on packed columns using SE-30, SKTFT-50X, and XE-60 as the stationary phase. The values of thermodynamic functions of sorption were calculated. The correlations between these functions and the molecular structures as well as the conditions of analysis were established. The insertion of the S atom into the molecules of derivatives of perfluorocarboxylic acids causes a decrease in the contribution of the orientation interaction and an increase in the dispersion interaction of thioesters with the stationary phases compared to esters and amides of perfluorocarboxylic acids studied previously.

**Key words:** perfluorocarboxylic thioesters, thermodynamic functions of sorption of homologous fragments; gas-liquid chromatography.

Previously, as part of systematic studies of the regularities of sorption of esters[2,3] and amides[4] of perfluorocarboxylic acids, the retention indices and thermodynamic functions of sorption were calculated. The perfluorocarbonyl group substantiallly affects the character of sorption of the compounds compared to nonfluorinated esters. It was demonstrated that the retention parameters increased nonadditively as the lengths of both the alkyl perfluoroalkyl chains increased. The largest deviations from the linearity were observed for the first three members of the homologous series. When perfluorocarboxylic thioesters were sorbed on the polar XE-60 and PEG-40M stationary phases, the inverse retention order of the members of the series was observed as the length of the perfluoroalkyl chain increased. Nonadditivity of the retention parameters depends on the conditions of analysis (the type of the stationary phase and the temperature) and on the nature of sorbates.[2,3]

For amides of perfluorocarboxylic acids, deviations from linearity and the possibility of inversion of the retention order are more pronounced than for esters. In going from esters to amides of perfluorocarboxylic acids, the differential molar free energy of sorption increases, which may be caused by the higher polarity of the amide functional group.[4]

The aim of this work was to obtain new data on the retention parameters of thioesters of perfluorocarboxylic acids on three stationary phases of different nature, to calculate the contributions of the $CH_2$ and $CF_2$ groups to the values of the differential molar free energy, molar enthalpy and entropy of sorption of these groups, and to establish the dependences of the thermodynamic functions of sorption on the molecular structures and the nature of the stationary phase.

## Experimental

Synthesis of thioesters of polyfluorocarboxylic acids, $R_FC(O)SR$ ($R_F = CF_3$, $C_2F_5$, $C_3F_7$, $C_5F_{11}$, or $C_6F_{13}$), was carried out without isolation of the products from the reaction mixture. Pyridine (0.25 mL) and the perfluoroacylating agent (0.50 mL) were added to a solution of thiol (10 mL) in *n*-hexane (the concentration was 1 mg $mL^{-1}$). The reaction mixture was kept for 30—50 min and washed with a saturated aqueous solution of $NaHCO_3$. The upper layer was separated and dried with $Na_2SO_4$. Solutions analyzed by GLC were diluted to the concentration of $10^{-3}$ mg $mL^{-1}$. In the chromatograms, the peak areas of impurities were no more than 3% of the peak areas of the compounds under study. The thioesters $C_4F_9C(O)SR$ were analyzed as solutions of pure samples.

The retention times were determined accurate to ±0.5 s. The studies were carried out on a Tsvet-550M chromatograph equipped with an electron capture detector and glass columns (3 m×1.5 mm) packed with 10% SKTFT-50X on Chromaton N AW-HMDS (0.16—0.20 mm), 10% SE-30 on Chromaton N AW-HMDS (0.16—0.20 mm), and 5% XE-60 on Chromaton N AW-DMCS (0.16—0.20 mm). Argon was used as the carrier gas. The flow rate was 30 mL $min^{-1}$. The thermostat temperature was 60—180 °C. The injector temperature was 120—220 °C. The detector temperature was 300 °C. The retention time of the slightly sorbed gas ($SF_6$) was taken as the "dead time" ($t_0$). The value of $t_0$ was monitored after each 20 assays.

---

[*] For Part 1, see Ref. 1.

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 6, pp. 1165—1168, June, 1997.

The values of the differential molar free energy of sorption ($\Delta G_{i,j}(CX_2)$) was calculated by the formula:

$$\Delta G_{i,j}(CX_2) = -RT\ln(t'_n/t'_{n-1}),$$

where $t'_n$ and $t'_{n-1}$ are corrected retention times of adjacent homologs, which differ by one $CX_2$ group (X = H or F). The values of the molar enthalpy $\Delta H(CX_2)$ and entropy $\Delta S(CX_2)$ of sorption of the $CX_2$ group were determined by the least-squares method. The errors in determination of $\Delta G_{i,j}(CX_2)$ were no more than 0.05 kJ $mol^{-1}$ for the SE-30 and SKTFT-50X stationary phases and 0.02 kJ $mol^{-1}$ for the XE-60 stationary phase. The dispersions of the coefficients $\Delta H_{i,j}$ and $\Delta S_{i,j}$ were no more than 0.05 kJ $mol^{-1}$ and 0.06 J $mol^{-1}$ K, respectively.

## Results and Discussion

Perfluorocarboxylic thioesters under study of the general formula $CF_3(CF_2)_jC(O)S(CH_2)_iCH_3$ ($i$ and $j$ = 0—5) were treated as six homologous series whose members differ by the $CH_2$ group with the fixed value of $R_F$ and six pseudohomologous series whose members differ by the $CF_2$ group with the fixed value of R. The calculated changes in the thermodynamic functions of sorption, which occur as the lengths of the alkyl and perfluoroalkyl chains increase, are given in Tables 1 and 2. However, when this formal approach is used, it should be remembered that the above-mentioned values reflect not only the contributions of the $CX_2$ groups but the changes in the interactions of the molecule with the stationary phase as well.

**Changes in the thermodynamic functions of sorption, which occur as the length of the alkyl chain increases.** The values of the contributions of $\Delta G_i(CH_2)$ of perfluorocarboxylic thioesters sorbed on three stationary phases are given in Table 1. The contribution to the sorption energy of the $CH_2$ groups (equal to 0.35—0.65 kJ $mol^{-1}$) most sharply increases when the first $CH_2$ group is added. When $i > 3$, $\Delta G_i(CH_2)$ have closer values for both homologous series as well as for various stationary phases. As the length of the alkyl chain increases, the effect of the perfluoroalkyl substituent is diminished. These regularities are common to all stationary phases under study.

The values of the contributions to the sorption energy decrease in the series $\Delta G_i(CH_2)_{SE-30}$ >

**Table 1.** Changes in the thermodynamic functions of sorption of the $CH_2$ groups ($\Delta G_i(CH_2)$/kJ $mol^{-1}$ (120 °C), $\Delta H_i(CH_2)$/kJ $mol^{-1}$, and $\Delta S_i(CH_2)$/J $mol^{-1}$ K) in the $R_FC(O)S(CH_2)_iCH_3$ molecules

| $R_F$ | $i$ | SE-30 | | | SKTFT-50X | | | XE-60 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | $-\Delta G_i$ | $-\Delta H_i$ | $-\Delta S_i$ | $-\Delta G_i$ | $-\Delta H_i$ | $-\Delta S_i$ | $-\Delta G_i$ | $-\Delta H_i$ | $-\Delta S_i$ |
| $CF_3$ | 1 | 1.22 | 2.93 | 4.38 | 1.33 | 2.80 | 3.85 | 0.92 | 2.76 | 4.75 |
| | 2 | 1.63 | 3.79 | 5.50 | 1.68 | 3.20 | 3.87 | 1.37 | 3.72 | 6.00 |
| | 3 | 1.78 | 3.96 | 5.75 | 1.70 | 3.47 | 4.50 | 1.56 | 3.91 | 6.00 |
| | 4 | 1.80 | 4.06 | 5.67 | 1.71 | 3.48 | 4.50 | 1.58 | 3.94 | 6.00 |
| | 5 | 1.84 | 4.15 | 5.83 | 1.72 | 3.49 | 4.50 | 1.61 | 3.97 | 6.00 |
| $C_2F_5$ | 1 | 1.16 | 2.78 | 3.96 | 1.16 | 2.69 | 3.75 | 0.81 | 2.71 | 4.90 |
| | 2 | 1.60 | 3.85 | 5.72 | 1.60 | 3.25 | 4.20 | 1.35 | 3.71 | 6.00 |
| | 3 | 1.74 | 3.90 | 5.50 | 1.65 | 3.40 | 4.40 | 1.48 | 3.85 | 6.00 |
| | 4 | 1.77 | 4.03 | 5.75 | 1.68 | 3.43 | 4.46 | 1.58 | 4.00 | 6.20 |
| | 5 | 1.82 | 4.16 | 6.00 | 1.69 | 3.46 | 4.50 | 1.60 | 3.98 | 6.00 |
| $C_3F_7$ | 1 | 1.21 | 2.70 | 3.70 | 1.16 | 2.64 | 3.75 | 0.74 | 2.70 | 4.80 |
| | 2 | 1.55 | 3.52 | 4.90 | 1.58 | 3.24 | 4.10 | 1.34 | 3.50 | 5.50 |
| | 3 | 1.72 | 3.77 | 5.40 | 1.66 | 3.35 | 4.30 | 1.47 | 3.83 | 6.00 |
| | 4 | 1.76 | 3.82 | 5.30 | 1.68 | 3.32 | 4.20 | 1.57 | 3.93 | 6.00 |
| | 5 | 1.80 | 3.92 | 5.62 | 1.69 | 3.39 | 4.35 | 1.59 | 3.95 | 6.00 |
| $C_4F_9$ | 1 | 1.20 | 2.59 | 3.56 | 1.17 | 2.40 | 3.20 | 0.73 | 2.58 | 4.75 |
| | 2 | 1.59 | 3.55 | 5.00 | 1.55 | 3.12 | 4.00 | 1.32 | 3.48 | 5.50 |
| | 3 | 1.70 | 3.72 | 5.15 | 1.62 | 3.23 | 4.00 | 1.46 | 3.82 | 6.00 |
| | 4 | 1.75 | 3.82 | 5.25 | 1.64 | 3.31 | 4.25 | 1.55 | 3.91 | 6.00 |
| | 5 | 1.78 | 3.87 | 5.25 | 1.65 | 3.30 | 4.20 | 1.58 | 3.94 | 6.00 |
| $C_5F_{11}$ | 1 | 1.18 | 2.56 | 3.50 | 1.17 | 2.30 | 2.90 | 0.72 | 2.49 | 4.50 |
| | 2 | 1.59 | 3.55 | 5.00 | 1.52 | 3.10 | 4.00 | 1.28 | 3.44 | 5.50 |
| | 3 | 1.69 | 3.65 | 5.00 | 1.58 | 3.23 | 4.20 | 1.45 | 3.72 | 5.75 |
| | 4 | 1.76 | 3.72 | 5.00 | 1.64 | 3.19 | 4.00 | 1.55 | 3.81 | 5.75 |
| | 5 | 1.76 | 3.71 | 5.00 | 1.63 | 3.20 | 4.00 | 1.57 | 3.92 | 6.00 |
| $C_6F_{13}$ | 1 | 1.18 | 2.55 | 3.50 | 1.10 | 2.10 | 2.50 | 0.70 | 2.30 | 3.80 |
| | 2 | 1.59 | 3.55 | 5.00 | 1.50 | 3.06 | 4.00 | 1.28 | 3.45 | 5.50 |
| | 3 | 1.67 | 3.71 | 5.00 | 1.50 | 3.13 | 4.00 | 1.43 | 3.71 | 5.75 |
| | 4 | 1.74 | 3.71 | 5.00 | 1.63 | 3.18 | 4.00 | 1.54 | 3.72 | 5.50 |
| | 5 | 1.76 | 3.70 | 5.00 | 1.61 | 3.00 | 3.90 | 1.56 | 3.72 | 5.50 |

**Table 2.** Changes in the thermodynamic functions of sorption of the $CF_2$ groups ($\Delta G_j(CF_2)$/kJ mol$^{-1}$ (at 120 °C), $\Delta H_j(CF_2)$/kJ mol$^{-1}$, and $\Delta S_j(CF_2)$/J mol$^{-1}$ K) in the $CF_3(CF_2)_jC(O)SR$ molecules

| R | *i* | SE-30 | | | SKTFT-50X | | | XE-60 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | $-\Delta G_j$ | $-\Delta H_j$ | $-\Delta S_j$ | $-\Delta G_j$ | $-\Delta H_j$ | $-\Delta S_j$ | $-\Delta G_j$ | $-\Delta H_j$ | $-\Delta S_j$ |
| $CH_3$ | 1 | 0.65 | 2.28 | 4.15 | 0.63 | 2.40 | 4.50 | 0.16 | 2.05 | 5.20 |
| | 2 | 0.99 | 2.38 | 3.50 | 1.07 | 3.03 | 5.00 | 0.48 | 2.25 | 4.49 |
| | 3 | 1.02 | 2.56 | 3.87 | 1.12 | 3.23 | 5.35 | 0.53 | 2.51 | 5.02 |
| | 4 | 1.02 | 2.77 | 4.45 | 1.18 | 3.24 | 5.25 | 0.62 | 2.39 | 4.51 |
| | 5 | 1.06 | 3.18 | 5.25 | 1.25 | 3.22 | 5.23 | 0.64 | 2.44 | 4.57 |
| $C_2H_5$ | 1 | 0.63 | 2.06 | 3.65 | 0.50 | 2.38 | 4.84 | −0.27 | 2.03 | 5.86 |
| | 2 | 0.96 | 2.32 | 3.40 | 1.06 | 3.02 | 5.00 | 0.47 | 2.20 | 4.38 |
| | 3 | 0.98 | 2.57 | 4.00 | 1.14 | 3.10 | 5.00 | 0.49 | 2.53 | 5.19 |
| | 4 | 0.99 | 2.77 | 4.50 | 1.19 | 3.15 | 5.00 | 0.57 | 2.46 | 4.83 |
| | 5 | 1.02 | 3.10 | 5.30 | 1.21 | 3.37 | 5.50 | 0.58 | 2.47 | 4.81 |
| $C_3H_7$ | 1 | 0.50 | 1.91 | 3.60 | 0.43 | 2.20 | 4.50 | −0.29 | 1.70 | 5.06 |
| | 2 | 0.94 | 2.61 | 3.79 | 1.03 | 3.00 | 5.00 | 0.43 | 2.13 | 4.31 |
| | 3 | 1.01 | 2.64 | 4.13 | 1.09 | 3.07 | 5.00 | 0.49 | 2.51 | 5.15 |
| | 4 | 1.03 | 2.95 | 5.00 | 1.17 | 3.12 | 4.95 | 0.55 | 2.42 | 4.76 |
| | 5 | 1.05 | 3.13 | 5.33 | 1.19 | 3.24 | 5.20 | 0.57 | 2.50 | 4.90 |
| $C_4H_9$ | 1 | 0.48 | 1.83 | 3.45 | 0.39 | 1.77 | 3.50 | −0.35 | 1.55 | 4.83 |
| | 2 | 0.92 | 2.41 | 3.79 | 1.00 | 2.97 | 5.00 | 0.39 | 2.10 | 4.30 |
| | 3 | 1.00 | 2.60 | 4.20 | 1.10 | 3.06 | 5.00 | 0.46 | 2.40 | 4.94 |
| | 4 | 1.00 | 2.82 | 4.69 | 1.15 | 3.11 | 5.00 | 0.54 | 2.34 | 4.60 |
| | 5 | 1.04 | 3.17 | 5.46 | 1.17 | 3.14 | 5.00 | 0.56 | 2.38 | 4.64 |
| $C_5H_{11}$ | 1 | 0.47 | 1.76 | 3.30 | 0.36 | 1.54 | 3.00 | −0.38 | 1.51 | 4.79 |
| | 2 | 0.90 | 2.30 | 3.50 | 0.97 | 2.74 | 4.50 | 0.38 | 2.11 | 4.44 |
| | 3 | 0.99 | 2.67 | 4.00 | 1.06 | 2.94 | 4.75 | 0.45 | 2.34 | 4.81 |
| | 4 | 0.97 | 2.78 | 4.45 | 1.14 | 3.10 | 5.00 | 0.51 | 2.24 | 4.40 |
| | 5 | 0.98 | 2.95 | 5.00 | 1.17 | 3.04 | 4.80 | 0.54 | 2.27 | 4.51 |
| $C_6H_{13}$ | 1 | 0.45 | 1.66 | 3.05 | 0.33 | 1.44 | 2.82 | −0.38 | 1.30 | 4.20 |
| | 2 | 0.90 | 2.44 | 4.00 | 0.96 | 2.56 | 4.10 | 0.36 | 2.07 | 4.37 |
| | 3 | 0.96 | 2.33 | 3.52 | 1.04 | 2.82 | 4.50 | 0.44 | 2.23 | 4.54 |
| | 4 | 0.98 | 2.65 | 4.30 | 1.13 | 2.95 | 4.64 | 0.50 | 2.21 | 4.35 |
| | 5 | 0.98 | 2.75 | 4.50 | 1.14 | 3.06 | 4.80 | 0.53 | 2.22 | 4.30 |

$\Delta G_i(CH_2)_{SKTFT\text{-}50X} > \Delta G_i(CH_2)_{XE\text{-}60}$. The exception is the $CF_3C(O)S(CH_2)_iCH_3$ compounds ($i = 0-2$) for which $\Delta G_i(CH_2)_{SKTFT\text{-}50X} > \Delta G_i(CH_2)_{SE\text{-}30}$, as was the case for the corresponding esters.[2,3]

The character of the changes in the thermodynamic functions $\Delta H$ and $\Delta S$ of sorption of the $CH_2$ group is the same for all stationary phases under study. The lowest values of $\Delta H_i(CH_2)$ and $\Delta S_i(CH_2)$ are observed in the case of $i = 1$. When $i > 3$, the parameters remain virtually unchanged for each stationary phase. Elongation of the perfluoroalkyl chain leads to a decrease in the thermodynamic functions of sorption of the $CH_2$ group of perfluorocarboxylic thioesters.

$\Delta H_i(CH_2)$ and $\Delta S_i(CH_2)$ for perfluorocarboxylic thioesters sorbed on the SE-30 and XE-60 stationary phases have close values. For perfluorocarboxylic thioesters with the same alkyl substituent, a slight decrease in the values of $\Delta H_i(CH_2)$ and $\Delta S_i(CH_2)$ was observed in going from the $CF_3$ to $C_6F_{13}$ derivatives. When $i = 1$, this decrease is more pronounced (see Table 1). For the series of perfluorocarboxylic thioesters, the values of $\Delta H_i(CH_2)$ and $\Delta S_i(CH_2)$ are higher than for perfluorocarboxylic esters. Apparently, the effect of the thiocarboxyl group on the adjacent $CH_2$ group is weaker than that of the carboxyl group. Elongation of the C—S bond compared to the C—O bond leads to a decrease in the barrier to free rotation of the adjacent alkyl groups and an increase in the energy of the dispersion interaction with a stationary phase.

**Changes in the thermodynamic functions that occur as the length of the perfluoroalkyl chain increases.** The values of $\Delta G_j(CF_2)$ are smaller than $\Delta G_i(CH_2)$ for all stationary phases (see Table 2). An increase in the length of the perfluoroalkyl chain leads to an increase in the values of $\Delta G_j(CF_2)$. The values of $\Delta G_j(CF_2)$ for the compounds with the identical $R_F$ decrease as the length of the alkyl chain increases. This change is most pronounced in the case of SKTFT-50X. When $j > 2$, the order $\Delta G_j(CF_2)_{SKTFT\text{-}50X} > \Delta G_j(CF_2)_{SE\text{-}30} > \Delta G_j(CF_2)_{XE\text{-}60}$ is true for all series. The authors[3] believed that this indicates that the interaction between the perfluoroalkyl chain and the fluorine-containing stationary phase is strengthened as the length of this chain increases.

The tendency of a change in the contributions of the $CF_2$ groups to the energy of sorption on XE-60 is analogous to that observed in the cases of SKTFT-50X and SE-30, but the absolute values of $\Delta G_j(CF_2)$ are substantially smaller (see Tables 1 and 2). Even when the fourth difluoromethylene group was inserted, the values of $\Delta G_j(CF_2)$ were approximately half the values for SE-30 and SKTFT-50X. The key feature of the interaction of perfluorocarboxylic thioesters with XE-60 is the inversion of the elution order of pseudohomologs: $C_2F_5 < CF_3 < C_3F_7$ when $j > 2$. The character of the temperature dependence of $\Delta G_j(CF_2)$ suggests that when the temperature of analysis ($T_a$) is >140 °C, pentafluoropropionic thioester will be eluted before trifluoroacetic thioester at any R. An increase of $T_a$ to 180 °C, should cause further changes in the elution order: $C_3F_7 < C_2F_5 < CF_3$. However in this case, highly efficient separation of the compounds with close values of the retention parameters should occur. An analogous inversion was observed for pseudohomologous series of perfluorocarboxylic esters as well as for unsubstituted and substituted amides of perfluorocarboxylic acids.[4] In the case of perfluorocarboxylic thioesters, a decrease in the value of $\Delta G$ for pseudohomologs containing the $C_2F_5$ substituent compared to the compounds containing the $CF_3$ substituent is more pronounced than that observed in the case of esters, *i.e.*, the values of $\Delta G$ for trifluoroacetates are higher than those for pentafluoropropionates. Apparently, three types of the derivatives of perfluorocarboxylic acids interact with the XE-60 stationary phase according to the same mechanism, whereas the quantitative ratios of the contributions of the dispersion, orientation, and induction forces are different.

The values of $\Delta H_j(CF_2)$ increase within each series of perfluorocarboxylic thioesters. The largest values of $\Delta S_j(CF_2)$ on the XE-60 stationary phase were observed in the case of $j = 1$. Then these values decreased owing to an increase in the lability of $CF_2$ groups. In the case of SE-30, the values of $\Delta S_j(CF_2)$ increase as the length of the perfluoroalkyl chain increases.

Comparatively close values of the contributions of the $CH_2$ and $CF_2$ groups to the differential molar free energy and the functional dependence of changes in the contributions to the thermodynamic functions of sorption on the molecular structures are the common characteristic features of interactions of esters and thioesters of perfluorocarboxylic acids with the stationary phases. The type of this dependence is retained for stationary phases with different polarity, which indicates that the dispersion interaction is the governing factor.

A number of peculiarities are determined by the presence of the S atom in molecules of perfluorocarboxylic thioesters. As the length of the alkyl chain increases in the homologous series in going from $i = 0$ to $i = 1$, the thermodynamic parameters of sorption change less sharply than in the series of perfluorocarboxylic esters.

Our studies allow us to make suggestions as to the nature of retention and the mechanism of sorption of the derivatives of perfluorocarboxylic acids and to reveal the role of the heteroatom in retention. When the O atom is replaced by the S atom, the C—S bond length increases, and the polarity of the molecule decreases. As a result, the mutual effect of the alkyl and perfluoroalkyl substituents is diminished, and the number of degrees of freedom of rotation of $CH_2$ groups increases. This leads to a decrease in the contribution of the orientation interaction and an increase in the dispersion interaction of the molecules of perfluorocarboxylic thioesters with the stationary phases compared to those of esters and amides of perfluorocarboxylic acids.

## References

1. V. E. Kirichenko, O. A. Osipova, and K. I. Pashkevich, *Izv. Akad. Nauk, Ser. Khim.*, 1997, 984 [*Russ. Chem. Bull.*, 1997, **46**, 944 (Engl. Transl.)].
2. E. P. Promyshlennikova, V. E. Kirichenko, K. I. Pashkevich, D. N. Grigor'eva, and R. V. Golovnya, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1991, 629 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1991, **40**, 547 (Engl. Transl.)].
3. E. P. Promyshlennikova, V. E. Kirichenko, K. I. Pashkevich, D. N. Grigor'eva, and R. V. Golovnya, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1991, 1487 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1991, **40**, 1312 (Engl. Transl.)].
4. V. E. Kirichenko, E. P. Promyshlennikova, and K. I. Pashkevich, *Izv. Akad. Nauk, Ser. Khim.*, 1994, 868 [*Russ. Chem. Bull.*, 1994, **43**, 812 (Engl. Transl.)].

*Received June 19, 1996;*
*in revised form December 10, 1996*