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SYNTHESIS OF ISOTHIOCOUMARIN DERIVATIVES

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A method was developed for the synthesis of 1-oxo-1H-isothiochromenes from 2-benzofuran-1(3H)-one (phthalide). 3-Bromo-6-chloro- and 3,6-dibromo-2-benzofuran-1(3H)-ones were prepared by the bromination of 6-chloro- and 6-bromo-2-benzofuran-1(3H)-ones and were converted by hydrolysis into 5-chloro- or 5-bromo-2-formylbenzoic acids. The condensation of these acids with rhodanine followed by recyclization gave 7-chloro- and 7-bromo-1-oxo-1H-isothiochromene-3-carboxylic acids.

Keywords: 2-benzofuran-1(3H)-one, 1-oxo-1H-isothiochromenone, 2-formylbenzoic acid, heterocyclization, recyclization.

Many derivatives of 1H-2-benzothiopyran (1H-isothiochromenes) exhibit biological activity [1-5]. However, due to the limited number of methods available for their synthesis the properties of these compounds have been insufficiently investigated [6]. 1H-2-Benzothiopyrans are produced by the reactions of 2-(aroylmethyl)benzoates with P_2S_5 [7], 2-cyanomethylbenzoates with carbon disulfide [8], and lithium N,N-diethyl-*o*-toluamide with thioethers [9]. Isothiochromene-1-thione was obtained with a yield of 12% by the reaction of N-cyanomethyl-N-methyl-2-chloro-5-nitrobenzamide with NaH and carbon disulfide in DMSO [10]. A more convenient method for the synthesis of isothiocoumarins is the reaction of *ortho*-formylbenzoic acids with rhodanine (2-thioxo-4-thiazolidinone) followed by recyclization of the obtained 5-arylidenerhodanine in a basic medium [11, 12] although the initial *ortho*-formylbenzoic acids are difficult to obtain. The possibilities of applying this method to the creation of combinatorial libraries of heterocyclic compounds with an isothiocoumarin fragment were demonstrated in [13]. The possibilities of the method are limited by the fact that the available starting compounds are formylbenzoic and opianic acids. During the production of substituted 2-formylbenzoic acids organometallic compounds are often used for formylation or carboxylation [14-16]. We note that the significant lability of the isothiocoumarin ring complicates the direct introduction of substituents by, for example, electrophilic substitution.

In the present work a convenient method is proposed for the synthesis of substituted isothiocoumarins of type **I** suitable for further transformations and for use in combinatorial chemistry. For this purpose derivatives of phthalide **III**, obtained by the reduction of phthalic anhydride, were used as precursors of 2-formylbenzoic acids **II**.

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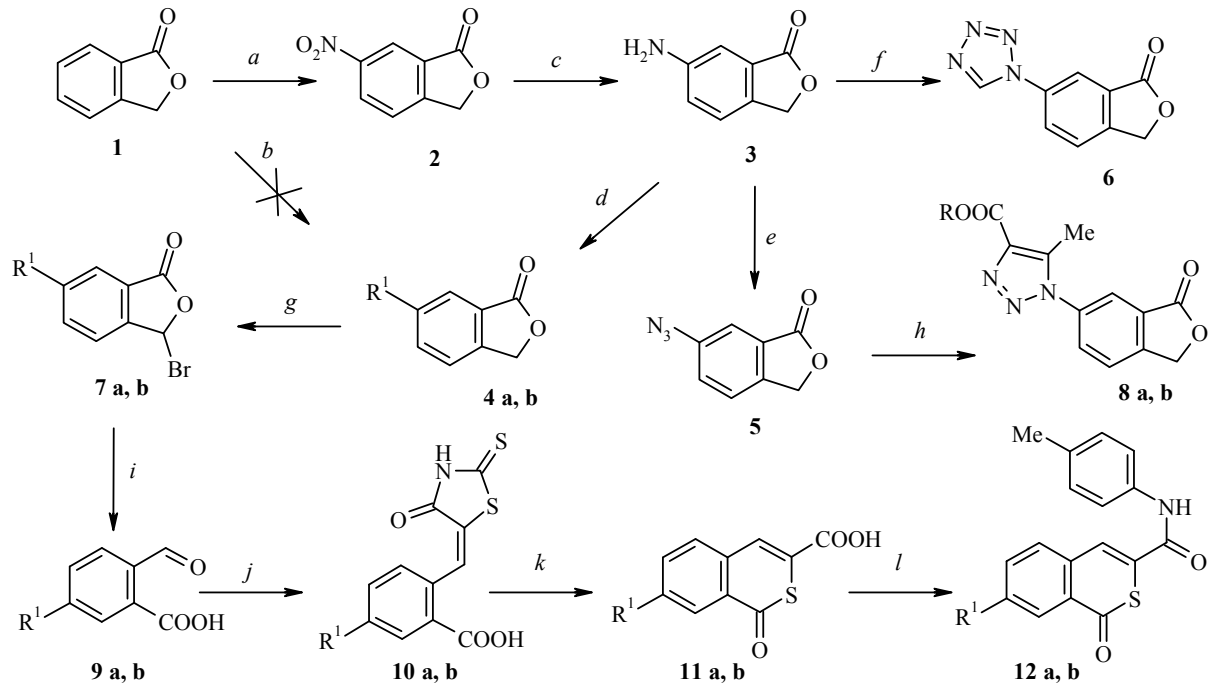
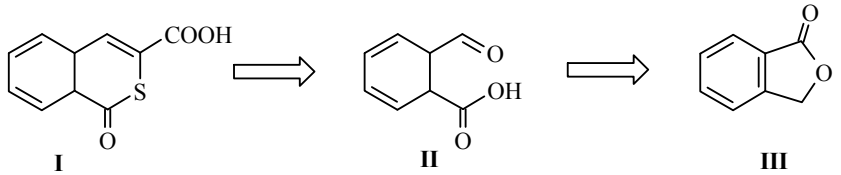


4, 7, 9-12 a R^1 = Cl, **b** R^1= Br; **8 a** R = H, **b** R = Me

Conditions and reagents: *a* KNO_3 in H_2SO_4; *b* 1.2 equiv. Br_2, 0.12 equiv. Fe, nitrotoluene, 135°C, 4 h; *c* Fe, EtOH–water, boiling 6 h (75%); *d* 1. $NaNO_2$, HCl/HBr. 2. CuCl/CuBr (74/71%); *e* 1. $NaNO_2$, HCl. 2. NaN_3 (78%); *f* 4.6 equiv. $CH(OEt)_3$, 1.2 equiv. NaN_3, AcOH, 100°C, 3 h (86%); g NBS, CCl_4, boiling 2 h; *h* For compound **8a**, acetoacetic ester, Na, MeOH (79%), (compounds **8a→8b**). 1. $SOCl_2$. 2. MeOH, 1 h (93%); *i* H_2O, boiling 3 h; *j* Rhodanine, AcOH, Et_3N (94/89%); *k* NaOH (64/61%); *l* l. $SOCl_2$, dioxane; 2. *p*-toluidine and Et_3N.



Attempts at the bromination of the phthalide **1** in the aromatic ring with bromine in the presence of $FeBr_3$ were unsuccessful; the reaction mixture resinifies under these conditions. We note that of electrophilic substitution reactions in the aromatic ring of phthalide only nitration has been described [17]. We reduced the nitrophthalide **2** formed in this reaction, and we converted the aminophthalide **3** into chloro(bromo)phthalides **4a**,**b** by the Sandmeyer reaction, into the azide **5** by diazotization and the subsequent action of sodium azide, and into the tetrazolylphthalide **6** by a three-component reaction with ethyl orthoformate and sodium azide by the procedure in [18].

Reactions involving bromination of the phthalides **2** and **4-6** at position 3 with N-bromosuccinimide were studied (Scheme). It was established that the reaction only takes place successfully in the case of compounds **4a**,**b**, resulting in the production of the phthalides **7a**,**b**. Bromination under these conditions is complicated by the poor solubility of compounds **2** and **6** in CCl_4 and oxidation of the azido group (compound **5**). Triazolecarboxylic acid **8a** and also the ester **8b** were obtained from the azide **5** by reaction with acetoacetic ester. Compounds **8a**,**b** are not brominated at position 3 by N-bromosuccinimide.

Formylbenzoic acids **9a**,**b** are formed during the hydrolysis of the phthalides **7a**,**b**. They are converted into isothiocoumarin-3-carboxylic acids **11a**,**b** in two stages by condensation with rhodanine and recyclization of the 5-arylidenerhodanines **10a**,**b**. In the case of the amides **12a**,**b** it was shown that the acids **11a**,**b** can be used in the synthesis of compounds with an isothiocoumarin fragment by acylation of the corresponding acid chlorides.

Thus, a method was developed for the synthesis of 1-oxo-1H-isothiochromene-3-carboxylic acids from substituted phthalides, and the limitations of this method in the case of certain substituents were revealed.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded in DMSO-d_6 on a Varian Unity +400 instrument (400 MHz) with TMS as standard. The mass spectra were obtained on an Agilent 1100 LC/MSD chromato-mass spectrometer with chemical ionization.

6-Amino-2-benzofuran-1(3H)-one (3). We heated 51.8 g of iron powder in 311 ml of a 3:1 mixture of ethanol and water with stirring. We added conc. HCl (6.2 ml), and slowly added the nitro derivative **2** (35.8 g, 200 mmol). The mixture was heated for 8 h, and the hot solution was quickly filtered. The amine released when the filtrate had cooled was filtered off and recrystallized. Yield 22.35 g (75%); mp 182°C (ethanol–water).

Synthesis of 6-Halo-2-benzofuran-1(3H)-ones 4a,b. The amine **3** (37.2 g, 250 mmol) was dissolved in conc. HCl (100 ml) or conc. HBr (190 ml). The mixture was cooled to 0°C, and a solution of $NaNO_2$ (17.2 g, 250 mmol) in the minimum amount of water was added dropwise with cooling. The temperature range of diazotization was 0-5°C. When necessary, the obtained solution of the diazonium salt was filtered. It was then added with stirring at 0°C to a previously prepared solution of copper(I) chloride (24.8 g, 250 mmol) in conc. HCl (50 ml) or copper(I) bromide (35.9 g, 250 mmol) in conc. HBr (90 ml). The reaction mixture was heated on a water bath. When the release of nitrogen had stopped, the solid product was filtered off and purified by recrystallization.

6-Chloro-2-benzofuran-1(3H)-one (4a). Yield 74%; mp 107-108°C (ethanol–water). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 5.36 (2H, s, CH_2); 7.67 (1H, d, ^3J = 8.8, H-4); 7.73 (1H, dd, ^3J = 8.8, ^4J = 2.0, H-5); 7.79 (1H, d, ^4J = 2.0, H-7). Mass spectrum, *m/z*: 169 $[M + H]^+$. Found, %: C 56.81; H 2.86. $C_8H_5ClO_2$. Calculated, %: C 57.00; H 2.99.

6-Bromo-2-benzofuran-1-(3H)-one (4b). Yield 71%; mp 114-115°C (ethanol–water). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 5.35 (2H, s, CH_2); 7.63 (1H, d, ^3J = 8.0, H-4); 7.70 (1H, dd, ^3J = 8.0, ^4J = 1.6, H-5); 7.78 (1H, d, ^4J = 1.6, H-7). Mass spectrum, *m/z*: 214 $[M + H]^+$. Found, %: C 45.22; H 2.19. $C_8H_5BrO_2$. Calculated, %: C 45.10; H 2.37.

6-Azido-2-benzofuran-1(3H)-one (5). The amine **3** (7.4 g, 50 mmol) was dissolved in conc. HCl (19 ml), the solution was cooled to 0°C, and a cooled solution of $NaNO_2$ (3.45 g, 50 mmol) in the minimum amount of water was added dropwise with stirring, while keeping the temperature below 5°C. When necessary, the obtained solution of 3-oxo-1,3-dihydroisobenzofuran-5-diazonium chloride was filtered and cooled to -5°C. A solution of NaN_3 (3.25 g, 50 mmol) in water (10 ml) was slowly added to it drop by drop with vigorous stirring while the temperature was kept below 7°C. After the addition of NaN_3, the mixture was kept at room temperature for 1 h. The azide **5** was filtered off, washed with a large volume of iced water to a neutral reaction, and dried in a dark cool place. Yield 6.8 g (78%); decomp. p. 127-128°C (ethanol–water). Mass spectrum, *m/z*: 176 $[M + H]^+$. Found, %: C 54.62; H 2.61; N 23.74. $C_8H_5N_3O_2$. Calculated, %: C 54.86; H 2.88; N 23.99.

6-(1H-Tetrazol-1-yl)-2-benzofuran-1(3H)-one (6). AcOH (40 ml) was added with stirring to a suspension of 5-aminophthalide **3** (0.74 g, 5 mmol) and NaN_3 (0.39 g, 6 mmol) in ethyl orthoformate (3.79 ml, 23 mmol). The mixture was heated at 95-100°C for 4 h. After cooling, 0.7 ml of conc. HCl was added, and the mixture was filtered. The filtrate was evaporated under vacuum, and the residue was recrystallized from ethanol.

Yield 1 g (86%); mp 216-218°C (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 5.49 (2H, s, CH_2); 7.93 (1H, d, ^3J = 8.8, H-4); 8.35 (1H, d, ^3J = 8.8, H-5); 8.46 (1H, s, H-7); 10.22 (1H, s, tetrazole). Mass spectrum, *m/z*: 203 [M + H]$^+$. Found, %: C 53.58; H 2.82; N 27.57. $C_9H_6N_4O_2$. Calculated, %: C 53.47; H 2.99; N 27.71.

Synthesis of 3-Bromo-6-halo-2-benzofuran-1(3H)-ones 7a,b. Phthalide **4** (10 mmol) was dissolved in CCl_4 (10 ml) that had been dried over P_2O_5. N-bromosuccinimide (1.78 g, 10 mmol) and then benzoyl peroxide (0.02 g) was added. The mixture was cautiously heated, and after the end of the reaction (the formation of succinimide on the surface), it was boiled for a further 10 min. After cooling, the succinimide was filtered off and washed with a small amount of CCl_4. The solvent was distilled from the combined filtrates under a weak vacuum on a water bath. The crystals were filtered off and recrystallized.

3-Bromo-6-chloro-2-benzofuran-1-(3H)-one (7a). Yield 1.9 g (77%); mp 57-58°C (hexane). Mass spectrum, *m/z*: 248 [M + H]$^+$. Found, %: C 38.74; H 1.48. $C_8H_4BrClO_2$. Calculated, %: C 38.83; H 1.63.

3,6-Dibromo-2-benzofuran-1(3H)-one (7b). Yield 2.36 g (81%); mp 64-65°C (hexane). Mass spectrum *m/z*: 293 [M + H]$^+$. Found, %: C 32.79; H 1.14. $C_8H_4Br_2O_2$. Calculated, %: C 32.91; H 1.38.

5-Methyl-1-(3-oxo-1,3-dihydro-2-benzofuran-5-yl)-1H-1,2,3-triazole-4-carboxylic Acid (8a). Sodium (0.3 g, 13 mmol) was dissolved in absolute ethanol (15 ml). To the cooled solution of sodium ethoxide, acetoacetic ester (1.75 g, 10 mmol) was added, and then azide **5** (1.75 g, 10 mmol) was slowly added (with cooling in iced water). The mixture was kept in an ice bath for 30 min and was then slowly heated to boiling and boiled for 1 h. A precipitate separated. Hot water was added to dissolve the precipitate (15-20 ml), and the mixture was boiled for a further hour. The hot solution was poured into 10 ml of conc. HCl and left to crystallize. The product was filtered off, washed on the filter with a small amount of water, and recrystallized. Yeld 2.05 g (79%); mp 207-208°C (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.58 (3H, s, CH_3); 5.51 (2H, s, CH_2); 7.93 (1H, d, *J* = 7.8, H-7); 7.98 (1H, d, *J* = 7.8, H-6); 8.06 (1H, s, H-4); 12.94 (1H, br. s, COOH). Mass spectrum, *m/z*: 260 [M + H]$^+$. Found, %: C 55.42; H 3.43; N 16.38. $C_{12}H_9N_3O_4$. Calculated, %: C 55.60; H 3.50; N 16.21.

Methyl 5-Methyl-1-(3-oxo-1,3-dihydro-2-benzofuran-5-yl)-1H-1,2,3-triazole-4-carboxylate (8b). A mixture of the acid **8a** (6.48 g, 25 mmol) and thionyl chloride (1.85 ml, 25 mmol) in dioxane (25 ml) was boiled for 1 h. The mixture was cooled, and the precipitated acid chloride was filtered off, washed with hexane, and added to methanol (15 ml). The reaction mixture was boiled for 1 h. The precipitate was filtered off, washed several times with water, and recrystallized from methanol. Yield 6.3 g (93%); mp 193-194°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.58 (3H, s, CH_3); 3.92 (3H, s, CH_3O); 5.52 (2H, s, CH_2); 7.93 (1H, d, *J* = 7.8, H-7); 7.98 (1H, d, *J* = 7.8, H-6); 8.08 (1H, s, H-4). Mass spectrum, *m/z*: 274 [M + H]$^+$. Found, %: C 57.42; H 3.92; N 15.24. $C_{13}H_{11}N_3O_4$. Calculated, %: C 57.14; H 4.06; N 15.38.

Synthesis of 2-Formyl-5-halobenzoic Acids 9a,b. A suspension of 3-bromobenzofuran **7a**,**b** (10 mmol) in water (5 ml) was heated for 3 h. The mixture was left in the refrigerator overnight, and the product was filtered off , washed with a small amount of cold ethanol, and dried in air.

5-Chloro-2-formylbenzoic Acid (9a). Yield 1.73 g (94%); mp 137-138°C (ethanol) [15].

5-Bromo 2-formylbenzoic Acid (9b), Yield 2.06 g (90%); mp 130-131°C (ethanol). Mass spectrum, *m/z*: 230 [M + H]$^+$. Found, %: C 42.07; H 2.09. $C_8H_5BrO_3$. Calculated, %: C 41.95; H 2.20.

Synthesis of 5-Halo-2-[(4-oxo-2-thioxo-1,3-thiazolidin-5-ylidene)methyl]benzoic Acids 10a,b. A mixture of aldehyde **9a**,**b** (10 mmol), rhodanine (1.3 g, 10 mmol), and triethylamine (2 ml) in acetic acid (25 ml) was boiled for 2 h. The product that formed on cooling was filtered off and was recrystallized after drying in air.

5-Chloro-2-[(4-oxo-2-thioxo-1,3-thiazolidin-5-ylidene)methyl]benzoic Acid (10a). Yield 2.8 g (94%); mp 261-262°C (AcOH). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.55 (1H, d, ^3J = 8.8, H-3); 7.69 (1H, d, ^3J = 8.8, ^4J = 2.0, H-4); 8.01 (1H, d, ^4J = 2.0, H-6); 8.15 (1H, s, CH=). Mass spectrum, *m/z*: 300 [M + H]$^+$. Found, %: C 44.01; H 2.16; N 4.42. $C_{11}H_6ClNO_3S_2$. Calculated, %: C 44.08; H 2.02; N 4.67.

5-Bromo-2-[(4-oxo-2-thioxo-1,3-thiazolidin-5-ylidene)methyl]benzoic Acid (10b). Yield 3.06 g (89%); mp 274-275°C (AcOH). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.46 (1H, d, ^3J = 8.8, H-3); 7.83 (1H, dd,

^3J = 8.8, ^4J = 2.0, H-4); 8.11 (1H, d, ^4J = 2.0, H-6), 8.17 (1H, s, CH=). Mass spectrum, *m/z*: 345 $[M+H]^+$. Found, %: C 38.24; H 1.51; N 4.01. $C_{11}H_6BrNO_3S_2$. Calculated, %: C 38.38; H 1.76; N 4.07.

Synthesis of 7-Halo-1-oxo-1H-isothiochromene-3-carboxylic acids 11a,b. Compound **10** (3.3 g, 10 mmol) was added to a solution of KOH (2.24 g, 40 mmol) in water (50 ml) and the mixture was boiled for 3 h. The reaction mixture was poured into conc. HCl (15 ml) and ice (75 g). The precipitate was filtered off and purified by recrystallization.

7-Chloro-1-oxo-1H-isothiochromene-3-carboxylic Acid (11a). Yield 1.54 g (64%); mp 273-274°C (ethanol–water). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.91 (1H, dd, ^3J = 7.8, ^4J = 2.0, H-6); 8.08 (1H, d, ^3J = 7.8, H-5); 8.16 (1H, s, H-4); 8.25 (1H, d, ^4J = 2.0, H-8). Mass spectrum, *m/z*: 241 $[M + H]^+$. Found, %: C 50.03; H 1.94. $C_{10}H_5ClO_3S$. Calculated, %: C 49.91; H 2.09.

7-Bromo-1-oxo-1H-isothiochromene-3-carboxylic Acid (11b). Yield 1.74 g (61%); mp 281-282°C (ethanol–water). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.97 (1H, d, ^3J = 7.8, H-5); 8.02 (1H, dd, ^3J = 7.8, ^4J = 2.0, H-6); 8.22 (1H, s, H-4); 8.27 (1H, d, ^4J = 2.0, H-8). Mass spectrum, *m/z*: 286 $[M + H]^+$. Found, %: C 42.29; H 1.54. $C_{10}H_5BrO_3S$. Calculated, %: C 42.13; H 1.77.

Synthesis of Amides 12a,b (General Method). A mixture of the acid **11a**,**b** (50 mmol) and thionyl chloride (3.7 ml, 50 mmol) in dioxane (50 ml) was boiled for 1 h. The mixture was cooled, and the precipitated acid chloride was filtered off and washed with hexane. To a solution of toluidine (0.59 g, 5.5 mmol) in dioxane, triethylamine (0.6 g, 5.5 mmol) and acid chloride **11a** or compound **11b** (5.5 mmol) was added. The reaction mixture was kept at room temperature for 2 h and was then heated nearly to boiling point. After cooling, it was diluted with water, and the precipitate was filtered off, washed several times with water, and recrystallized.

N-(4-Methylphenyl)-7-chloro-1-oxo-1H-isothiochromene-3-carboxamide (12a). Yield 1.72 (95%); mp 270-271°C (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.33 (3H, s, CH_3); 7.13 (2H, d, ^3J = 7.8, H-3,5 Ar); 7.57 (2H, d, ^3J = 7.8, H-2,6 Ar); 7.90 (1H, dd, ^3J = 8.8, ^4J = 2.0, H-6); 7.94 (1H, d, ^3J = 8.8, H-5); 8.15 (1H, d, ^4J = 2.0, H-8); 8.27 (1H, s, H-4); 10.43 (3H, s, NH). Mass spectrum, *m/z*: 330 $[M + H]^+$. Found, %: C 61.80; H 3.84; N 4.11. $C_{17}H_{12}ClNO_2S$. Calculated, %: C 61.91; H 3.67; N 4.25.

N-(4-Methylphenyl)-7-bromo-1-oxo-1H-isothiochromene-3-carboxamide (12b). Yield 2 g (97%); mp 277-278°C (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.33 (3H, s, CH_3); 7.13 (2H, d, ^3J = 7.8, H-3,5 Ar); 7.57 (2H, d, ^3J = 7.8, H-2,6 Ar); 7.85 (1H, d, ^3J = 8.8, H-5); 8.04 (1H, dd, ^3J = 8.8, ^4J = 2.0, H-6); 8.25 (1H, s, H-4); 8.29 (1H, d, ^4J = 2.0, H-8); 10.43 (3H, s, NH) . Mass spectrum, *m/z*: 375 $[M + H]^+$. Found, %: C 54.70; H 3.06; N 3.89. $C_{17}H_{12}BrNO_2S$. Calculated, %: C 54.56; H 3.23; N 3.74.

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