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Reaction of 2-Mono- and 2,2-Disubstituted 1,3-Dioxolanes with 1-Ethyl-3-hexylaluminacyclopentane

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Abstract — Reaction of 2-mono- and 2,2-disubstituted 1,3-dioxolanes with 1-ethyl-3-hexylaluminacyclopentane involves cleavage of the Al–C bond of the metal cycle and formation, after hydrolysis, ethylene glycol monoethers. Catalytic amounts of $ZrCl_4$ or $Ni(acac)_2$ drive the reaction with 2-monosubstituted 1,3-dioxolanes.

Aluminum-containing five-membered heterocycles (aluminacyclopentanes) [1] whose convenient synthesis has been developed fairly recently [2, 3] are highly reactive compounds. Their reaction with carbonyl substrates has yet poorly been explored. We recently found [4] that orthoformates selectively cleave 3-alkyl-substituted aluminacyclopentanes by the sterically less hindered endocyclic bond Al–C^5. Proceeding with this research, we brought 1-ethyl-3-hexylaluminacyclopentane (**I**) into reaction with 1,3-dioxolanes **IIa**–**IIe** whose cleavage with the simplest trialkylalanes [$AlEt_3$ or $Al(Bu\text{-}i)_3$] gives rise to higher ethylene glycol monoethers [5]. In the case of aluminacyclopentanes with three differently active Al–C bonds of which the most active are endocyclic [3], three different monoethers **III**–**V** can form from each acetal.

We found that the reaction studied involves predominantly the endocyclic radical of aluminacyclopentane, and the yield and ratio of cleavage products are much affected by the degree of substitution of the C^2 atom of the dioxolane ring. Thus 2,2-disubstituted 1,3-dioxolanes **IIa**, **IIb** react with a double excess of aluminacyclopentane, selectively providing, in mild conditions (20°C, 2 h), aluminates that convert to 2-hydroxy ethers **IIIa**, **IIIb** in yields of up to ~40% after hydrolysis (see table). At equimolar reactant ratios, the yield of the latter reaction products is no higher than 3–5%, whereas further increase of the excess of organoaluminum compound has no appreciable effect on the reaction result. The structure of the resulting compounds suggests that, like in the reaction with orthoformates [4], compound **I** regioselectively reacts by the sterically less hindered endocyclic bond Al–C^5. When the reaction time is increased to 6 h, along with ethers **IIIa**, **IIIb**, compounds **Va**, **Vb** are formed via cleavage of the exocyclic Al–C_2H_5 bond in yields of no higher than 15%. Replacement of an aliphatic solvent, hexane, by CH_2Cl_2 or Et_2O or raising the temperature to 60°C has almost no effect on the reaction yield and products, whereas using $ZrCl_4$ or $Ni(acac)_2$ that are known to catalyze reactions of cyclic acetals with $AlEt_3$ or $Al(Bu\text{-}i)_3$ [5], adversely affect the yield of ethers **IIIa**, **IIIb** because of formation of unidentified macromolecular compounds. It will be remembered that ordinary trialkylalanes [$AlEt_3$ or $Al(Bu\text{-}i)_3$] fail to react with cyclic acetals in ethers [5], whereas halohydrocarbons, by contrast, drive the reaction [6].

In similar conditions, 1-ethyl-3-hexylaluminacyclopentane less selectively reacts with 2-monosubstituted 1,3-dioxolanes **IIc**–**IIe** to form a mixture of heteroring cleavage products **IIIc**–**IIIe** and **IVc**–**IVe** in a ratio depending both on the structure of the starting acetals and on the reaction conditions (see table). Thus the above-mentioned metal-complex catalysts exert a marked effect of the regioselectivity of the process, the effect being the strongest (especially with $ZrCl_4$) with acetal **IId**. The effect of methylene chloride is ambiguos: It increases the total yield of **IIIc** and **IVc** with 2-phenyl-1,3-dioxolane (**IIc**) but has no effect on regioselectivity, whereas with 2-isopropyl-1,3-dioxolane (**IId**), it increases regioselectivity and has almost effect on the total yield of **IIId** and **IVd**.

It is interesting to note that compound **I** reacts with unsymmetrical acetal **IIe** by the dioxolane C^2–O^1 bond only, giving rise to roughly equal amounts of

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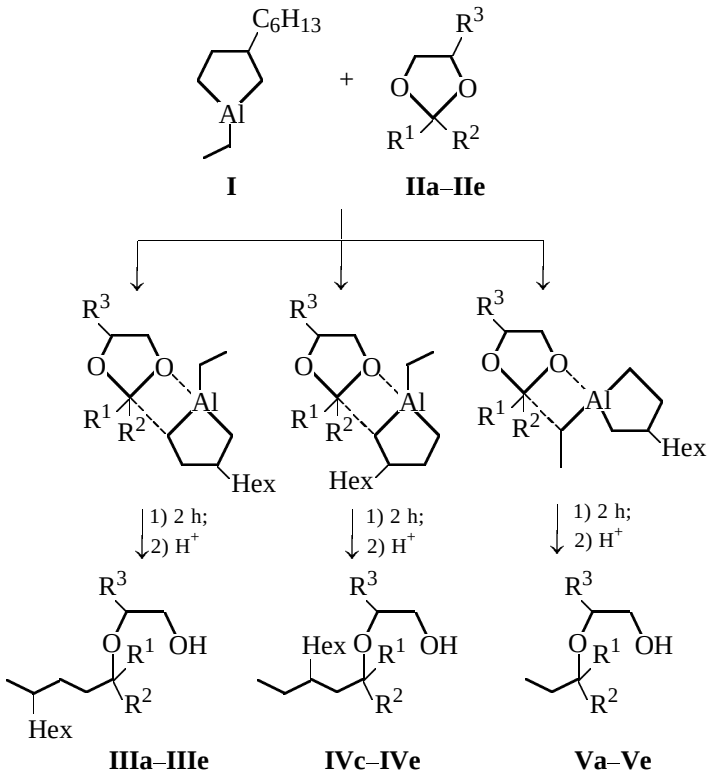

II–**V**, $R^1 = R^2 = Me$, $R^3 = H$ (**a**); $R^1 = R^2 = Et$, $R^3 = H$ (**b**); $R^1 = Ph$, $R^2 = R^3 = H$ (**c**); $R^1 = i\text{-Pr}$, $R^2 = R^3 = H$ (**d**); $R^1 = Ph$, $R^2 = Me$, $R^3 = H$ (e).

hydroxyethers **IIIe**, **IVe**, whereas monoethers with a secondary OH group were not detected among the reaction products. Therewith, aluminacyclopentane **I** proved to cleave the least regioselectively (see table). Monoethers **Vc**–**Ve** always formed only if the reaction time was increased to 6 h, but their yields were no higher than 5–10%.

Raising the temperature to 60°C slightly increased the total yield of hydroxyethers **IIIc**, **IIId** and **IVc**, **IVd**, but adversely affected the selectivity of formation of compounds **IIIc**, **IIId**.

Unsubstituted 1,3-dioxolane failed to react under our conditions.

The structure of the all products was established by ^1H and ^{13}C NMR spectroscopy. The spectra of compounds **IIIc**–**IIe** and **IVc**–**IVe** show doubled signals of certain carbon atoms because of diastereomer formation.

Thus, the reaction of 1,3-dioxolanes with 1-ethyl-3-hexylaluminacyclopentane can serve as a convenient synthetic route to 2-hydroxyethyl ethers of branched secondary and tertiary higher alcohols, which are hardly accessible by other methods. The effect of activating additives on the regioselectivity of the reaction and the yield of the target reaction products is not unambiguous and depends on the nature and position of substituents in the starting acetals.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were taken in $CDCl_3$ on a Bruker AM-300 spectrometer at 300.13 and 75.47 MHz, respectively. The ^{13}C NMR spectra were measured in the JMODCH mode and then calculated by additive schemes [7, 8]. Gas chromatography was performed on Chrom-5, stainless-steel column 120 × 0.5 cm, packing 5% SE-30 on Chromaton N-AW; flame ionization detector, carrier gas helium.

Aluminacyclopentane (**I**) was obtained by the procedure in [2]. 2-Mono- and 2,2-disubstituted 1,3-dioxolanes **IIa**–**IIe** were synthesized by the procedure in [9], distilled under argon over NaOH, and stored in an inert atmosphere. Their physicochemical constants were consistent with those reported in [10].

Reaction of 1-ethyl-3-hexylaluminacyclopentane (I) with 1,3-dioxolanes IIa–IIe. Dioxolane **IIa**–**IIe**, 20 mmol, was added dropwise under dry argon to a solution of 40 mmol of compound **I** in 10 ml of dry heptane at –20°C. The mixture was heated to a spe-

Reaction of 2-mono- and 2,2-disubstituted 1,3-dioxolanes with 1-ethyl-3-hexylaluminacyclopentane (reaction time 2 h, aluminacyclopentane : acetal = 2 : 1)

Starting acetal	Solvent	Catalyst (3 mol%)	Temperature, °C	Total yield of reaction products, %	**III/IV** ratio
IIa	Hexane	–	20	**IIIa** (40)	–
IIa	"	–	60	**IIIa** (48)	–
IIa	CH_2Cl_2	–	20	**IIIa** (40)	–
IIb	Hexane	–	20	**IIIb** (43)	–
IIb	"	–	60	**IIIb** (46)	–
IIb	CH_2Cl_2	–	20	**IIIb** (45)	–
IIc	Hexane	–	20	**IIIc** + **IVc** (44)	1.59
IIc	"	$ZrCl_4$	20	**IIIc** + **IVc** (71)	1.73
IIc	"	–	60	**IIIc** + **IVc** (64)	1.13
IIc	"	$Ni(acac)_2$	20	**IIIc** + **IVc** (77)	2.71
IIc	CH_2Cl_2	–	20	**IIIc** + **IVc** (61)	1.44
IIc	CH_2Cl_2	$ZrCl_4$	20	**IIIc** + **IVc** (75)	2.75
IId	Hexane	–	20	**IIId** + **IVd** (52)	1.36
IId	"	$ZrCl_4$	20	**IIId** + **IVd** (54)	5.00
IId	"	–	60	**IIId** + **IVd** (65)	1.24
IId	"	$Ni(acac)_2$	20	**IIId** + **IVd** (62)	1.95
IId	CH_2Cl_2	–	20	**IIId** + **IVd** (54)	1.45
IId	CH_2Cl_2	$ZrCl_4$	20	**IIId** + **IVd** (43)	2.58
IIe	Hexane	–	20	**IIIe** + **IVe** (65)	1.03
IIe	"	$ZrCl_4$	20	**IIIe** + **IVe** (64)	1.00

cified temperature and stirred for 2 or 6 h, after which it was cooled to –20°C and decomposed with 20 ml of 10% HCl. The organic layer was separated, dried with $MgSO_4$, and analyzed by GLC. Reaction products were isolated by vacuum duistillation. Hydroxyethers **IIIa** and **IIIb** were identified individual, **IIIc**–**IIIe**, as mixtures with their regioisomers **IV**. Compounds **Va**–**Ve** were identified by GLC using reference samples [6].

2-(1,1,4-Trimethyldecyloxy)ethan-1-ol (IIIa), bp 174°C (45 mm Hg), n_D^{20} 1.4553. ^{1}H NMR spectrum, δ, ppm: 0.86 d (3H, H^{11}, $CHCH_3$, *J* 6.2 Hz), 0.87 t (3H, H^{10}, CH_3, *J* 6.7 Hz), 1.08–1.14 m (2H, H^9, CH_2), 1.16 s (6H, $H^{1'}$, $H^{1''}$, CH_3), 1.20–1.57 m (13H, H^2–H^8, CH_2, CH), 2.17 br.s (1H, OH), 3.41 t (3H, $H^{1'''}$, CH_3, *J* 4.9 Hz), 3.63 t (3H, $H^{2'''}$, CH_2, *J* 4.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.00 (C^{10}, CH_3), 19.61 (C^{11}, $CHCH_3$), 22.58 (C^9, CH_2), 25.46 ($C^{1'}$, $C^{1''}$, CH_3), 26.95 (C^6, CH_2), 29.57 (C^7, CH_2), 30.79 (C^3, CH_2), 31.85 (C^8, CH_2), 33.16 (C^4, CH), 36.89 (C^5, CH_2), 37.45 (C^2, CH_2), 62.07 ($C^{2'''}$, CH_2OH), 62.18 ($C^{1'''}$, CH_2O), 74.92 (C^1, C). Found, %: C 74.33; H 13.20. $C_{15}H_{32}O_2$. Calculated, %: C 73.71; H 13.20.

2-(1,1-Diethyl-4-methyldecyloxy)ethan-1-ol (IIIb), bp 168°C (40 mm Hg) n_D^{20} 1.4794. ^{1}H NMR spectrum, δ, ppm: 0.85 d (3H, H^{11}, $CHCH_3$, *J* 5.9 Hz), 0.89 t (3H, H^{10}, CH_3, *J* 6.1 Hz), 1.01–1.75 m (25H, H^2–H^9, $H^{1'}$–$H^{5'}$, CH_2, CH), 2.33 br.s (1H, OH), 3.35 t (3H, $H^{1''}$, CH_2O, *J* 4.8 Hz), 3.70 t (2H, $H^{2''}$, CH_2, *J* 4.8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.14 (C^{10}, CH_3), 19.79 (C^{11}, $CHCH_3$), 21.96 ($C^{2''}$, $C^{4'}$, CH_2), 22.74 (C^9, CH_2), 26.01 ($C^{3'}$, CH_2), 27.10 (C^6, CH_2), 29.47 (C^7, CH_2), 29.71 (C^3, CH_2), 31.98 (C^8, CH_2), 33.32 (C^4, CH), 34.24 (C^2, CH_2), 34.47 ($C^{1'}$, $C^{5'}$, CH_2), 37.07 (C^5, CH_2), 60.75 ($C^{2''}$, CH_2OH), 62.47 ($C^{1''}$, CH_2O), 75.14 (C^1, C). Found, %: C 68.01; H 12.10. $C_{18}H_{36}O_2$. Calculated, %: C 67.88; H 12.03.

2-(4-Methyl-1-phenyldecyloxy)ethan-1-ol (IIIc), bp 120–125°C (1 mm Hg). ^{1}H NMR spectrum, δ, ppm: 0.88 d (3H, H^{11}, $CHCH_3$, *J* 6.7 Hz), 0.9 t (3H, H^{10}, CH_3, *J* 6.0 Hz), 1.15–1.45 m (11H, H^4–H^9, CH_2, CH), 1.65–1.75 m (2H, H^3, CH_2), 1.75–1.95 m (2H, H^2, CH_2), 2.17 br.s (1H, OH), 3.43 t (2H, $H^{1''}$, CH_2O, *J* 7.7 Hz), 3.70 t (2H, $H^{2''}$, CH_2OH, *J* 7.7 Hz), 4.20 t (1H, H^1, CH, *J* 7.0 Hz), 7.20–7.40 m (5H, $H^{2'}$–$H^{6'}$, CH, Ar). ^{13}C NMR spectrum, δC, ppm: 13.99 (C^{10}, CH_3), 19.46 (C^{11}, $CHCH_3$), 22.55 (C^9, CH_2), 29.49 (C^7, CH_2), 29.65 (C^6, CH_2), 31.77 (C^8, CH_2), 32.58 (C^4, CH), 32.67 (C^5, CH_2), 35.61 (C^2, CH_2), 36.73 (C^3, CH_2), 61.79 ($C^{2''}$, CH_2OH), 69.75 ($C^{1''}$, CH_2O), 83.15

(C^1, CH), 126.48–128.28 ($C^{2'}$–$C^{6'}$, CH, Ar), 142.42 ($C^{1'}$, C, Ar). Found, %: C 78.10; H 11.00. $C_{19}H_{32}O_2$. Calculated, %: C 78.03; H 10.94.

2-(3-Ethyl-1-phenylnonyloxy)ethan-1-ol (IVc), bp 120–125°C (1 mm Hg). ^{1}H NMR spectrum, δ, ppm: 0.84 t (3H, H^{11}, $CHCH_3$, *J* 5.1 Hz), 0.86 t (3H, H^9, CH_3, *J* 6.0 Hz), 1.15–1.45 m (10H, H^4–H^8, CH_2), 1.45–1.55 m (2H, H^{10}, CH_2), 1.65–1.75 m (2H, H^2, CH_2), 1.75–1.95 m (1H, H^3, CH), 2.17 br.s (1H, OH), 3.43 t (2H, $H^{1''}$, CH_2O, *J* 7.7 Hz), 3.70 t (2H, $H^{2''}$, CH_2OH, *J* 7.7 Hz), 4.32 t (1H, H^1, CH, *J* 6.8 Hz), 7.20–7.40 m (5H, $H^{2'}$–$H^{6'}$, CH, Ar). ^{13}C NMR spectrum, δ_C, ppm: 10.45 (C^{11}, CH_3), 13.99 (C^9, CH_3), 22.55 (C8, CH_2), 26.03 (C^5, CH_2), 26.84 (C^{10}, CH_2), 29.54 (C^6, CH_2), 31.77 (C^7, CH_2), 35.06 (C^3, CH), 35.47 (C^4, CH_2), 42.19 (C^2, CH_2), 61.79 (C2", CH_2OH), 69.75 ($C^{1''}$, CH_2O), 80.90–80.98 (C^1, CH), 126.48–128.28 ($C^{2'}$–$C^{6'}$, CH, Ar), 142.34 ($C^{1'}$, C, Ar). Found, %: C 78.10; H 11.00. $C_{19}H_{32}O_2$. Calculated, %: C 78.03; H 10.94.

2-(1-Isopropyl-4-methyldecyloxy)ethan-1-ol (IIId), bp 145–150°C (40 mm Hg). ^{1}H NMR spectrum, δ, ppm: 0.89 t (3H, H^{10}, CH_3, *J* 6.6 Hz), 0.9 d (3H, H^{11}, $CHCH_3$, *J* 5.2 Hz), 0.91 d (6H, $H^{2'}$, $H^{3'}$, CH_3, *J* 6.7 Hz), 1.05–1.55 m (15H, H^2–H^9, CH_2, CH), 1.80–1.92 m (1H, $H^{1'}$, CH), 2.25 br.s (1H, OH), 2.97–3.07 m (1H, H^1, CH), 3.55 t (2H, $H^{1''}$, CH_2O, *J* 7.0 Hz), 3.72 t (2H, $H^{2''}$, CH_2OH, *J* 7.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.06 (C^{10}, CH_3), 18.03 ($C^{2'}$, CH_3), 18.23 ($C^{3'}$, CH_3), 19.60, 19.71 (C^{11}, $CHCH_3$), 22.65 (C^9, CH_2), 27.01 (C^6, CH_2), 27.72 (C^2, CH_2), 29.63 (C^7, CH_2), 30.65 (C^4, CH), 31.90 (C^8, CH_2), 32.76, 32.86 (C^3, CH_2), 32.90, 33.01 ($C^{1'}$, CH), 36.90 (C^5, CH_2), 62.25 ($C^{2''}$, CH_2OH), 70.70 ($C^{1''}$, CH_2O), 85.37, 85.47 (C^1, CH). Found, %: C 73.36; H 13.13. $C_{16}H_{34}O_2$. Calculated, %: C 74.36; H 13.26.

2-(3-Ethyl-1-isopropylnonyloxy)ethan-1-ol (IVd), bp 145–150°C (40 mm Hg). ^{1}H NMR spectrum, δ, ppm: 0.87 t (3H, H^{11}, $CHCH_3$, *J* 6.7 Hz), 0.91 d (6H, $H^{2'}$, $H^{3'}$, CH_3, *J* 6.7 Hz), 0.92 t (3H, H^9, CH_3, *J* 6.9 Hz), 1.05–1.55 m (15H, H^2–H^8, H^{10}, CH_2, CH), 1.80–1.92 m (1H, $H^{1'}$, CH), 2.25 br.s (1H, OH), 3.13–3.21 m (1H, H^1, CH), 3.55 t (2H, $H^{1''}$, CH_2O, *J* 7.0 Hz), 3.72 t (2H, $H^{2''}$, CH_2OH, *J* 7.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 10.30, 10.78 (C^{11}, $CHCH_3$), 14.06 (C^9, CH_3), 17.66, 17.83 ($C^{3'}$, CH_3), 18.13, 18.20 ($C^{2'}$, CH_3), 23.02 (C^8, CH_2), 26.23 (C^5, CH_2), 27.01 (C^{10}, CH_2), 29.63 (C^6, CH_2), 30.61, 30.65 (C^3, CH), 31.90 (C^7, CH_2), 33.72, 34.30 (C^4, CH_2), 35.59 ($C^{1'}$, CH), 37.08 (C^2, CH_2), 62.25 ($C^{2''}$, CH_2OH), 70.47 ($C^{2'}$, CH_2O), 82.80 (C^1, CH_2). Found, %: C 73.36; H 13.13. $C_{16}H_{34}O_2$. Calculated, %: C 74.36; H 13.26.

2-(1,4-Dimethyl-1-phenyldecyloxy)ethan-1-ol (IIIe), bp 180–185°C (5 mm Hg). ^{1}H NMR spectrum, δ, ppm: 0.85 t (3H, H^{10}, CH_3, *J* 7.0 Hz), 0.90 d (3H, H^{11}, $CHCH_3$, *J* 5.7 Hz), 1.13 d (3H, $H^{3''}$, CH_3, *J* 5.8 Hz), 1.15–1.95 m (15H, H^2–H^9, CH_2, CH), 2.11 br.s (1H, OH), 3.45 t (2H, $H^{2''}$, CH_2, *J* 5.2 Hz), 3.50–3.60 m (1H, $H^{1''}$, CHO,), 7.20–7.40 m (5H, $H^{2'}$–$H^{6'}$, CH, Ar). ^{13}C NMR spectrum, δ_C, ppm: 14.06 (C^{10}, CH_3) 17.56 ($C^{3''}$, CH_3), 19.55 (C^{11}, $CHCH_3$), 22.64 (C^9, CH_2), 29.57 (C^7, CH_2), 29.71 (C^6, CH_2), 31.86 (C^8, CH_2), 32.75 (C^4, CH), 35.91 (C^5, CH_2), 36.04 (C^2, CH_2), 36.80 (C^3, CH_2), 65.65 ($C^{2''}$, CH_2OH), 74.27 ($C^{1''}$, CHO), 81.71 (C^1, CH), 126.60–128.49 ($C^{2'}$–$C^{6'}$, CH, Ar), 143.98 ($C^{1'}$, C, Ar). Found, %: C 78.40; H 12.01. $C_{20}H_{34}O_2$. Calculated, %: C 78.38; H 11.18.

2-(1,4-Dimethyl-1-phenyldecyloxy)ethan-1-ol (IVe), bp 180–185°C (5 mm Hg). ^{1}H NMR spectrum, δ, ppm: 0.85 t (3H, H^{11}, CH_3, *J* 7.1 Hz), 0.95 t (3H, H^9, CH_3, *J* 6.6 Hz), 1.13 d (3H, $H^{3''}$, CH_3, *J* 5.8 Hz), 1.15–1.95 m (15H, H^2–H^8, H^{10}, CH_2, CH), 2.11 br.s (1H, OH), 3.49 t (2H, $H^{2''}$, CH_2, *J* 6.6 Hz), 3.50–3.60 m (1H, $H^{1''}$, CHO,), 7.20–7.40 m (5H, $H^{2'}$–$H^{6'}$, CH, Ar). ^{13}C NMR spectrum, δ_C, ppm: 10.53 (C^{11}, CH_3), 14.06 (C^9, CH_3), 17.64 ($C^{3''}$, CH_3), 22.64 (C^8, CH_2), 26.05, 26.37 (C^{10}, CH_2) 26.11 (C^5, CH_2), 29.66 (C^6, CH_2), 31.86 (C^7, CH_2), 35.21 (C^3, CH), 35.91 (C^4, CH_2), 42.71, 42.77 (C^2, CH_2), 65.75 ($C^{2''}$, CH_2, OH), 74.45 ($C^{1''}$, CHO), 79.49 (C^1, CH), 126.60–128.49 ($C^{2'}$–$C^{6'}$, CH, Ar), 143.90 ($C^{1'}$, C, Ar). Found, %: C 78.40; H 12.01. $C_{20}H_{34}O_2$. Calculated, %: C 78.38; H 11.18.

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