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THE FORMATION AND ALKYLATION OF (TRIMETHYLSILYL)-, (PHENYLDIMETHYLSILYL)-, AND (DIPHENYLMETHYLSILYL)-DICHLOROMETHYLLITHIUM. PREPARATION OF α,α-DICHLOROALKYLSILANES AND ((*Z*)-1-CHLORO-1-ALKENYL)-SILANES

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Summary

(Dichloromethyl)trimethylsilane, Me_3SiCCl_2H, (dichloromethyl)phenyldimethylsilane, $PhMe_2SiCCl_2H$, and (dichloromethyl)diphenylmethylsilane, $Ph_2MeSiCCl_2H$, readily react with lithium diisopropylamide in THF at —78°C to form the corresponding α-silyldichloromethyllithium reagents. The relative stability of the lithium reagents at —78° C is $Ph_2MeSiCCl_2Li \sim PhMe_2SiCCl_2Li >> Me_3SiCCl_2Li$. All three of the lithium reagents are readily silylated with chlorosilanes or alkylated with primary bromides and iodides. The α,α-dichloroalkylsilanes formed from the alkylation are stereoselectively dehydrochlorinated in refluxing dimethylformamide to ((*Z*)-1-chloro-alkenyl)silanes.

Introduction

In our research on the preparation of carbo-functional organosilanes via organoboranes [1], we prepared trimethylsilyldichloromethyllithium (I) according to Seyferth [2] and treated it with trialkylboranes at temperatures between —100° and —78° C (eq. 1). Reagent I has been shown to be unstable above —78° C and is best used at —100° C [3].

$$Me_3SiCCl_2H \xrightarrow[THF/-100°C]{\text{n-BuLi}} \underset{(I)}{Me_3SiCCl_2Li} \xrightarrow[(2)\,H_2O_2/HO^-]{(1)\,R_3B} Me_3SiCR_2 \qquad (1)$$

The low temperatures necessary resulted in slow or poor reactions of reagent I with organoboranes and this prompted us to investigate (phenyldimethylsilyl)-

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dichloromethyllithium (II) and (diphenylmethylsilyl)dichloromethyllithium (III) as possible alternatives to I.

$PhMe_2SiCCl_2Li$ (II) $Ph_2MeSiCCl_2Li$ (III)

Villieras et al. [4] have formed (trimethylsilyl)dibromomethyllithium (IV) by treatment of (dibromomethyl)trimethylsilane with lithium diisopropylamine (LDA) (eq. 2). We decided to employ this approach to reagents I, II, and III and to investigate their relative stabilities (eq. 3).

$$Me_3SiCBr_2H \xrightarrow[-78^\circ C]{LDA/THF} Me_3SiCBr_2Li \quad (2)$$

$$Ph_nMe_{3-n}SiCCl_2H \xrightarrow[-78^\circ C]{LDA/THF} Ph_nMe_{3-n}SiCCl_2Li \quad (3)$$

Results and discussion

Thermal stability of Me_3SiCCl_2Li, $PhMe_2SiCCl_2Li$ and $Ph_2MeSiCCl_2Li$

In our studies it was found that a principal mode of decomposition of these reagents is the self-coupling reaction illustrated in eq. 4. This type of coupling has also been noted in other work [2,3]. For example when I was formed at

$$RMe_2SiCCl_2Li + RMe_2SiCCl_2H \rightarrow RMe_2SiCCl_2SiMe_2R \quad (4)$$

R = Ph, Me

−78° C and quenched within two minutes with triethylchlorosilane at that temperature, a 93% yield (by NMR) of (trimethylsilyl)(triethylsilyl)dichloromethane was obtained indicating a high yield of I. When the reagent was not quenched within two minutes, the solution turned dark brown and eventually black and the yield decreased. This is consistent with the observations of Seyferth and coworkers [2,3]. When the solution of I was allowed to warm slowly to room temperature and was quenched with water (or triethylchlorosilane), the only isolated product was bis(trimethylsilyl)dichloromethane. This is best explained by an equilibrium involving the lithium reagent (I) and diisopropylamine to form (dichloromethyl)trimethylsilane (V) and LDA. Lithium reagent I then reacts with V to give the observed product (eq. 5 and 6). In order to rule out the formation of trimethylsilylchlorocarbene as an intermedi-

$$Me_3SiCCl_2Li + i\text{-}Pr_2NH \rightleftharpoons \underset{(V)}{Me_3SiCCl_2H} + LDA \quad (5)$$

$$Me_3SiCCl_2Li + Me_3SiCCl_2H \rightarrow (Me_3Si)_2CCl_2 + LiCHCl_2 \quad (6)$$

ate in the reaction, a 1 *M* solution of I in THF was formed in the presence of a 5-fold excess of cyclohexene and allowed to warm to room temperature. Again the only isolated product was the self-coupled product, bis(trimethylsilyl)dichloromethane, with no evidence for 7-chloro-7-(trimethylsilyl)-norcarane. This is consistent with the results of Olofson et al. [5] who have observed trimethylsilylcarbene in hexane solvent, but not in THF.

It was felt that the use of the phenyldimethylsilyl or diphenylmethylsilyl group rather than the trimethylsilyl group might add to the stability of the lithium reagent and at the same time decrease the rate of displacement of $CHCl_2^-$. Reagent II was readily formed upon treatment of (dichloromethyl)phenyldimethylsilane with LDA in THF for 1 h at —78° C. II was formed considerably more slowly than I, as evidenced by the presence of 40% of the starting material after 10 min at —78° C (trimethylchlorosilane quench). When the solution of II was quenched with trimethylchlorosilane after 1 h a 95% yield (by NMR) of (phenyldimethyl)(trimethylsilyl)dichloromethane was observed. Though formed more slowly than I, reagent II is much more stable, surviving several hours at —78° C without noticeable decomposition or loss of activity. The reagent II also is much more stable at temperature above —78° C. However, it retained only 70% of its activity at —50° C. Reagent II also undergoes the self-coupling reaction as its principal mode of decomposition (eq. 7 and 8). Thus, when II was formed at —78° C and allowed to warm to room temperature, a

$$PhMe_2SiCCl_2Li + i\text{-}Pr_2NH \rightleftharpoons PhMe_2SiCCl_2H + LDA \quad (7)$$

$$PhMe_2SiCCl_2Li + PhMe_2SiCCl_2H \rightarrow (PhMe_2Si)_2CCl_2 + LiCHCl_2 \quad (8)$$

40% yield of bis(phenyldimethylsilyl)dichloromethane was isolated. Strong evidence for the equilibrium illustrated in eq. 7 comes from the fact that when a 2-fold excess of LDA was employed in the formation of the lithium reagent II and the solution quenched with trimethylchlorosilane at —30° C, a quantitative yield (by NMR) of (phenyldimethylsilyl)(trimethylsilyl)dichloromethane was observed. If this solution was allowed to warm to 25° C before adding the trimethylchlorosilane, only the self-coupling product (eq. 8) was observed.

Reagent III was formed quantitatively from (dichloromethyl)diphenylmethylsilane and LDA at —78° C in 1 h and is very stable at that temperature. Quenching of III with trimethylchlorosilane at —78° C gave (diphenylmethylsilyl)(trimethylsilyl)dichloromethane in 95% yield (by NMR). A similar quench at —60° C gave a 72% yield of $Ph_2MeSiCCl_2SiMe_3$ and 31% yield of (dichloromethyl)diphenylmethylsilane. When the quench was carried out at 25° C, a 40% yield of the starting silane was obtained with none of the self-coupling or the desired product observed. Thus, the addition of one or two phenyl groups to the silyl function increases considerably the stability of the silyldichloromethyllithium reagent and the presence of two phenyl groups eliminates the self-coupling reaction.

Alkylations of Me_3SiCCl_2Li, $PhMe_2SiCCl_2Li$ and $Ph_2MeSiCCl_2Li$

All of these reagents (I, II, and III) generated according to eq. 3, could be silylated with chlorosilanes (eq. 9) or alkylated with primary bromides or iodides (eq. 10) at —78° C in good to excellent yield. The lithium reagent (I) failed to react with benzyl chloride, the self-condensation reaction being faster than the alkylation. Reagent I, however, reacted quite nicely with benzyl bromide to give the desired alkylated product in 48% yield. Attempts to alkylate I, II or III with secondary halides such as 2-bromopropane or bromocyclohexane, even in the presence of potassium iodide and HMPA, or with added TMEDA, resulted in no alkylation and observed instead were the formation of self-

condensation products in the case of reagents I and II and the recovery of starting material in the case of reagent III. A rigorous check by GLC for cyclohexene in the reactions of II or III with bromocyclohexane showed that elimination is not a side-reaction, at least in this secondary system. Table 1 gives the results of the alkylations and silylations of I, II and III. These reactions present excellent entries into the bis-silyldichloromethane derivatives and α,α-dichloroalkylsilanes, both of which are capable of further elaboration.

$$Ph_nMe_{3-n}SiCCl_2Li \xrightarrow{Me_3SiCl} Ph_nMe_{3-n}SiCCl_2SiMe_3 \quad (9)$$

$$Ph_nMe_{3-n}SiCCl_2Li \xrightarrow{RCH_2X} Ph_nMe_{3-n}SiCCl_2CH_2R \quad (10)$$

(VI)

The preparation of ((Z)-1-chloro-1-alkenyl)silanes

In view of the continued interest in the chemistry of α-halovinylsilanes [6,7] we decided to investigate the dehydrochlorination of the α,α-dichloroalkylsilanes (VI). A wide variety of bases including LDA, n-BuLi, t-BuOK, KOH and pyridine gave mixed results, leading to partial elimination, attack on silicon

(continued on p. 19)

TABLE 1

SILYLATION AND ALKYLATION OF α-SILYLDICHLOROMETHYLLITHIUM REAGENTS

R_3SiCCl_2Li [a]	RX (mmol)	Product	Yield	
			NMR [b]	Isolated
I	Me_3SiCl (11)	$(Me_3Si)_2CCl_2$	70	69
I	Et_3SiCl (11)	$Me_3SiCCl_2SiEt_3$	93	85
I	CH_3I (50)	$Me_3SiCCl_2CH_3$	93	80
I	CH_3CH_2Br (11)	$Me_3SiCCl_2CH_2CH_3$	98	84
I	$CH_3CH_2CH_2CH_2Br$ (11)	$Me_3SiCCl_2CH_2CH_2CH_2CH_3$	93	75
I	$PhCH_2Br$ (11)	$Me_2SiCCl_2CH_2Ph$	—	48
I	$PhCH_2Cl$ (11)	[c]	—	— [c]
II	Me_3SiCl (35)	$PhMe_2SiCCl_2SiMe_3$	—	78
II	$PhMe_2SiCl$ (20)	$(PhMe_2Si)_2CCl_2$	—	77
II	CH_3I (20)	$PhMe_2SiCCl_2CH_3$	—	81
II	CH_3CH_2Br (11)	$PhMe_2SiCCl_2CH_2CH_3$	—	64
II	$CH_3CH_2CH_2CH_2Br$ (20)	$PhMe_2SiCCl_2CH_2CH_2CH_2CH_3$	—	78
II	$Ch_2{=}CHCH_2Br$ (20)	$PhMe_2SiCCl_2CH_2CH{=}CH_2$	—	78
II	$PhCH_2Cl$ (20)	$PhMe_2SiCCl_2CH_2Ph$	—	69
II	C—$C_6H_{11}Br$ (20)	[d]	—	— [d]
III	Me_3SiCl (20)	$Ph_2MeSiCCl_2SiMe_3$	95	87
III	CH_3I (20)	$Ph_2MeSiCCl_2CH_3$	92	85
III	CH_3CH_2Br (20)	$Ph_2MeSiCCl_2CH_2CH_3$	93	90
III	$CH_3CH_2CH_2CH_2Br$ (20)	$Ph_2MeSiCCl_2CH_2CH_2CH_2CH_3$	—	67
III	$PhCH_2Cl$ (20)	$Ph_2MeSiCCl_2CH_2Ph$	63	58
III	$CH_3CH_2CH_2Cl$ (20)	none [e]		
III	C—$C_6H_{11}Br$ (20)	none [e]		
III	$(CH_3)_2CHBr$	none [e]		

[a] 10 mmol of lithium reagent. [b] Determined by adding CH_2Br_2 or CH_2Cl_2 as an internal standard to crude reaction residue. [c] Only $(Me_3Si)_2CCl_2$ isolated. [d] Only $(PhMe_2Si)_2CCl_2$ isolated. [e] Only $Ph_2MeSiCHCl_2$ isolated.

TABLE 2

CHARACTERIZATION OF α,α-DICHLOROSILANES

Product	B.p. (°C/mmHg)	$n_D(T)$	NMR [a] (δ, ppm)
$Me_3SiCCl_2SiMe_3$	90—95/80	1.4646(22) [b]	0.23 (s)
$Me_3SiCCl_2SiEt_3$	101—107/10	1.4848(22)	0.24 (s, 9H); 1.38—0.44 (m, 15H)
$Me_3SiCCl_2CH_3$	m.p. 115.5—117°C [c]	—	0.23 (s, 9H); 2.03 (s, 3H)
$Me_3SiCCl_2CH_2CH_3$	68—73/73	1.4553(22)	0.25 (s, 9H); 1.22 (t, 3H, *J* 7 Hz); 2.08 (q, 2H, *J* 7 Hz)
$Me_3SiCCl_2CH_2CH_2CH_2CH_3$	97—99/20	1.4585(21)	0.24 (s, 9H); 2.28—0.73 (m, 9H)
$Me_3SiCCl_2CH_2Ph$	m.p. 55—56°C	—	0.36 (s, 9H); 3.48 (s, 2H); 7.30 (s, 5H)
$PhMe_2SiCCl_2SiMe_3$	99—101/0.1	1.5266(24)	—0.01 (s, 9H); 0.54 (s, 6H); 7.63—7.97 (m, 5H)
$PhMe_2SiCCl_2SiMe_2Ph$	165—180/0.15 m.p. 58.5—59°C	—	0.23 (s, 12H); 7.65—7.03 (m, 10H)
$PhMe_2SiCCl_2CH_3$	81—85/0.1	1.5345(22)	0.52 (s, 6H); 1.93 (s, 3H); 7.70—7.05 (m, 5H)
$PhMe_2SiCCl_2CH_2CH_3$	100—110/0.4	1.5330(21)	0.56 (s, 6H); 1.12 (t, 3H, *J* 7.5 Hz); 2.0 (q, 2H, *J* 7.5 Hz; 7.60—7.00 (m, 5 H)
$PhMe_2SiCCl_2CH_2CH_2CH_2CH_2$	125—130/1.25	1.5239(22)	0.54 (s, 6H); 2.15—0.69 (m, 9H); 7.71—7.10 (m, 5H)
$PhMe_2SiCCl_2CH_2Ph$	150—160/0.10 m.p. 81—82	1.5715(25)	0.49 (s, 6H); 3.22 (s, 2H); 7.73—6.92 (m, 10H)
$PhMe_2SiCCl_2CH_2CH{=}CH_2$	112—115/0.20	1.5357(22)	0.59 (s, 6H); 2.75 (d, 2H, *J* 6.5 Hz); 6.25—5.58 (m. 3H) 7.72—7.00 (m, 5H)
$Ph_2MeSiCCl_2SiMe_3$	159—160/0.12	1.5697(21)	—0.03 (s, 9H); 0.77 (s, 3H); 7.83—7.04 (m, 10H)
$Ph_2MeSiCCl_2CH_3$	148—150/0.15	1.5870(20)	0.78 (s, 3H); 2.03 (s, 3H); 7.73—6.88 (m, 10H)
$Ph_2MeSiCCl_2CH_2CH_3$	144—146/0.04	1.5851(21)	0.79 (S, 3H); 1.15 (5, 3H, *J* 6.5 Hz); 2.11 (q, 2H, *J* 6.5 Hz) 7.70—7.00 (m, 10H)
$Ph_2MeSiCCl_2CH_2CH_2CH_2CH_3$	148—150/0.02	1.5720(21)	0.79 (s, 3H); 2.25—0.70 (m, 9H); 7.68—7.01 (m, 10H)
$Ph_2MeSiCCl_2CH_2Ph$	m.p. 71.5—73°C		0.69 (s, 3H); 3.32 (s, 2H); 7.73—6.95 (m, 15H)

[a] In CCl_4, with respect to internal TMS. [b] Lit. n_D^{25} 1.4667; ref. 2,3. [c] Lit. m.p. 117—119°C; ref. 2,3.

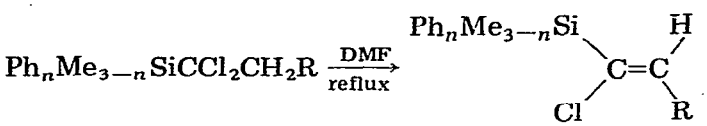
TABLE 3

YIELDS AND PROPERTIES OF ((*Z*)-1-CHLORO-1-ALKYL)SILANES

Silane	% Yield [a]	B.p. (°C/mmHg) (m.p. (°C))	$n'_D(T)$	NMR
$PhMe_2SiCCl{=}CH_2$	73	71—73/1.5	1.5235(22)	7.53—7.07 (m, 5H), 5.90 (s, 1H), 5.54 (s, 1H), 0.48 (s, 6H)
$PhMe_2SiCCl{=}CHCH_3$	80	91—93/1.5	1.5339(21)	7.47—6.95 (m, 5H), 5.77 (t, 1H), *J* 6 Hz), 1.74 (d, 3H, *J* 6 Hz), 0.42 (s, 6H)
$PhMe_2SiCCl{=}CHCH_2CH_2CH_3$	77	114—120/2.0	1.5183(22)	7.48—6.95 (m, 5H), 5.77 (t, 1H, *J* 7 Hz), 2.17 (q, 2H, *J* 7 Hz), 1.8—0.7 (m, 5H), 0.43 (s, 6H)
$PhMe_2SiCCl{=}CHPh$	85	146—148/0.3	1.5882(21)	7.90—7.70 (m, 5H), 6.65 (s, 1H), 0.53 (s, 6H)
$Ph_2MeSiCCl{=}CH_2$	60	130—135/0.05	1.5819(21)	7.53—6.95 (m, 10H), 6.00 (s, 1H, *J* 6 Hz), 1.78 (d, 3H, *J* 6 Hz), 0.67 (s, 3H)
$Ph_2MeSiCCl{=}CHCH_3$	83	(72—73)	—	7.50—6.95 (m, 10H), 5.86 (q, 1H, *J* 6 Hz), 1.78) d, 3H, *J* 6 Hz), 0.67 (s, 3H)
$Ph_2MeSiCCl{=}CHCH_2CH_2CH_3$	78	137—138/0.05	1.5675(21)	7.53—6.97 (m, 10H), 5.81 (t, 1H, *J* 6.5 Hz), 2.28 (q, 2H, *J* 7 Hz), 1.81—0.57 (m, 7H), 0.69 (s, 3H)
$Ph_2MeSiCCl{=}CHPh$	79	[b]	1.5704(21)	7.66—6.82 (m, 15H), 6.73 (s, 1H), 0.77 (s, 3H)

[a] Isolated yields. [b] Final purification by GLC.

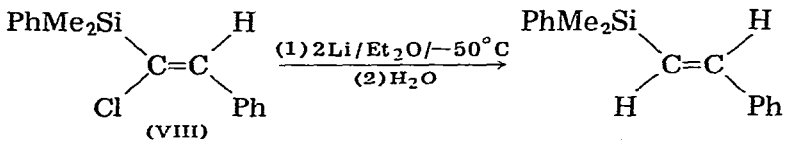
and, in general, poor yields of the desired α-chlorovinylsilane. It was subsequently discovered that the loss of HCl could be achieved by heating compounds VI and HMPA. This reaction was rather slow and not clean. However, refluxing the compounds VI in *N,N*-dimethylformamide resulted in a clean conversion to the α-chlorovinylsilanes. The reaction is very highly stereoselective giving only the ((*Z*)-1-chloro-1-alkenyl)silane VII (eq. 11).



(11)

(VII)

Three pieces of evidence strongly support the assigned stereochemistry. First of all when ((*Z*)-β-chloro-β-(phenyldimethylsilyl)styrene (VIII) was treated with lithium dispersion in ether at —50°C followed by quenching with water a 60% yield of *trans*-β-(phenyldimethylsilyl)styrene [8] was obtained (eq. 12). Secondly, the



(12)

(VIII)

chemical shifts of the vinyl protons in all the α-chlorovinylsilanes (VII) are consistent with those of Zweifel and Lewis for ((*Z*)-1-chloro-1-alkenyl)trimethylsilanes and about 0.5 ppm upfield from the values reported for the *E* isomers [6]. Finally, when ((*Z*)-1-chloro-1-(phenyldimethylsilyl)propene was irradiated in methylene chloride with a 250 Watt sun lamp in the presence of small amounts of bromine and pyridine only the starting α-chlorovinylsilane was isolated (90%) with no evidence for isomerization. Zweifel and Lewis [6] have shown that ((*E*)-1-chloro-1-alkenyl)trimethylsilanes isomerize to the *Z* isomers in high yield and high isomeric purity under these conditions.

It is important to note that the trimethylsilyl systems VI ($n = 0$) gave poor yields of the α-chlorovinylsilane (VII) and considerable decomposition. The phenylated systems VI ($n = 1$ or 2) however, underwent facile dehydrochlorination.

Considering the ease of preparing the α,α-dichloroalkylsilanes (VI) and the stereoselective, high yield dehydrochlorination to VII, this sequence offers a convenient stereoselective approach to ((*Z*)-1-chloro-1-alkenyl)silanes from readily available starting materials. The yields, and physical and NMR spectral properties of the ((*Z*)-1-chloro-1-alkenyl)silanes are given in Table 3.

Experimental

General comments

All reactions were conducted in flame-dried systems under a nitrogen atmosphere. The standard apparatus consisted of a three-necked, round-bottomed flask equipped with magnetic stirrer, condenser and septum inlet. The products were distilled at reduced pressure after removal of the solvent by distillation. A

final purification using preparative gas-liquid chromatography was employed in most cases. All compounds show IR, NMR, and mass spectral data in complete accord with the assigned structures.

Infrared spectra were recorded on a Perkin—Elmer 237 spectrophotometer, NMR spectra on a Varian T-60 or a Perkin—Elmer R-24 spectrophotometer and mass spectra on a Varian Mat CH-7. Tetrahydrofuran was donated by Pfizer of Puerto Rico and was distilled from sodium-benzophenone prior to use. (Dichloromethyl)dimethylchlorosilane was purchased from Petrarch Systems and converted to (dichloromethyl)trimethylsilane [9] and (dichloromethyl)phenyldimethylsilane by treatment with methylmagnesium iodide (76%) and phenylmagnesium bromide (85%), respectively. In a like manner, (dichloromethyl)-methyldichlorosilane (Petrarch) was converted to (dichloromethyl)diphenylmethylsilane with two equivalents of phenylmagnesium bromide (58%). Trimethylchlorosilane, a gift from Bristol of Puerto Rico, was distilled prior to use. Phenyldimethylchlorosilane was prepared according to Bamford and Pant [10]. All other compounds were commercially available and were distilled prior to use.

Characterizing physical and spectral data for the compounds reported here are given in Table 2.

Preparation of α,α-dichlorosilanes. General procedure

The standard apparatus (100 ml capacity) was charged with 20 ml of THF and 1.11 g (11 mmol) of diisopropylamine. This solution was cooled to 0°C and 11 mmol of n-butyllithium in hexane added over 1 min. After it had been stirred for 15 min at 0°C, the solution was cooled to —78°C (Dry Ice acetone) and 10 mmol of the appropriate dichloromethylsilane in THF added. The lithium reagent was allowed to form over an appropriate length of time (2 min for I, 1 h for II and III) and then from 11—50 mmol of the chlorosilane or alkyl halide added. After it had been stirred at —78°C for 30 min, the solution was hydrolyzed by the addition of 20 ml of 1.5 *N* HCl, saturated with sodium chloride and extracted with ether (3 × 20 ml). The organic layer was dried (Na_2SO_4) and concentrated by fractional distillation and the product distilled at reduced pressure.

Preparation of ((Z)-1-chloro-1-alkenyl)silanes. General procedure

A 25 ml, round-bottomed flask was charged with from 4—8 mmol of the α,α-dichloroalkylsilane and ca. 15 ml of dry dimethylformamide. The solution was refluxed with stirring for 16 h, cooled and diluted with 50 ml of pentane. This solution was washed with 1.5 *N* HCl (3 × 25 ml) and water (3 × 25 ml). After the organic layer was dried (Na_2SO_4), filtered and concentrated, the residue was distilled through a micro-distillation apparatus.

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