A One-Pot In-situ Formation and Reaction of Trimethyl(trichloromethyl)silane: Application to the Synthesis of 2,2,2-Trichloromethylcarbinols

Kevin E. Henegar*and Ricardo Lira

Pfizer Global Research and Development, Groton Laboratories Eastern Point Road, Groton CT 06340

kevin.e.henegar@pfizer.com

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- ¹³C NMR Spectrum of 7e S45
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- ¹H NMR Spectrum of 7f S48
- ¹³C NMR Spectrum of 7f S49
- ¹H NMR Spectrum of 8f S50
- ¹³C NMR Spectrum of 8f S51
- ¹H NMR Spectrum of 7g S52
- ¹³C NMR Spectrum of 7g S53
- ¹H NMR Spectrum of 8g S54
- ¹³C NMR Spectrum of 8g S55
- ¹H NMR Spectrum of 7h S56
- ¹³C NMR Spectrum of 7h S57
- ¹H NMR Spectrum of 8h S58
- ¹³C NMR Spectrum of 8h S59

General Methods

Materials were obtained from commercial suppliers and used as received. Tetra *n*-butylammonium acetate was 97% purity grade. NMR spectra were run on commercial spectrometers operating at 400 MHz for ¹H and 100 MHz for ¹³C. The chemical shifts are given in parts per million (ppm) relative to residual CDCl₃ at δ 7.27 ppm for proton spectra and δ 77.00 ppm for carbon spectra, or relative to residual DMSO at δ 2.50 for proton spectra and δ 39.7 for carbon spectra. IR spectra were recorded on an FT-IR spectrometer as thin films deposited on a diamond crystal (single bounce-diamond crystal (SB-DC)). Mass spectra were obtained using high and low resolution GC mass spectrometers. Melting points were recorded with open capillaries and are uncorrected. Silica chromatography was performed with silica gel grade 60 (230-400 mesh).

CCl₃-TMS

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ate	10 Aug 2011 08	:45:52		Date Stamp	10 Aug 2011 08	45:52	
ile Name	\\SEG3\SAMBA	2\BH010310\KEHENEGA\100	82011-KEHENEGA	PDATA\1\1r		Frequency (MHz)	400.13
ucleus	1H	Number of Transients	16	Origin	spect	Original Points Cour	t 32768
wner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	32.34
W(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.1736	Spectrum Type	STANDARD
weep Width (Hz)	8223.56	Temperature (degree C) 22.175				
1.0- 0.9- 0.8- 0.7-				7.27			
16 1	5 14	13 12 11 10	9 8	7 6 5 Chemical Shift (ppm)	4 3	2 1 0	-1 -2 -3 -4

1	0.37	148.8	1.0000
2	7.27	2908.9	0.0224

CCl₃-TMS

(sec)	1.3631	Comment	703478x167 trichlon	omethyltrimethylsilane 130	C day CDCI3 /opt/topsp	in3.0/data kehenega	
ate	10 Aug 2011 08:5	6:32		Date Stamp	10 Aug 2011 08:56:3	2	
ile Name	ISEG3ISAMBA2	BH010310/KEHENEGA/1008	2011-KEHENEGA\2\	PDATA\1\1r	1999/04/2014/549 2014/56/2014	Frequency (MHz)	100.61
lucleus	13C	Number of Transients	256	Origin	spect	Original Points Count	32768
)wner	nmr	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	184.55
W(cyclical) (Hz)	24038.46	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10061.3105	Spectrum Type	STANDARD
weep Width (Hz)	24037.73	Temperature (degree C) 23.526				
10082011-KEHE 1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 0.1 0.1	NEGA.00209tical	₩aleFactor = 1			76.68		-3.87

No.	(ppm)	(Hz)	Height
1	-3.87	-389.1	1.0000
2	76.68	7714.9	0.0923
3	77.00	7747.2	0.0918
4	77.31	7778.7	0.0914
5	95.40	9598.0	0.0318









Acquisition Time (sec)	3.9846	Comment	mostly wrong diasteremer from 703478x169 filtrate PROTON CDCl3 /opt/topspin3.0/data kehenega						
Date	06 Oct 2011 09:34:5	56		Date Stamp	06 Oct 2011 09:34	:56			
File Name	\\SEG3\SAMBA2\BH	010310/KEHENEGA/0610	2011-KEHENEGA\2\		Frequency (MHz)	400.13			
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768		
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	82.63		
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.2988	Spectrum Type	STANDARD		
Sweep Width (Hz)	8223.56	Temperature (degree C	24.996						





Acquisition Time (sec)	3.0409	Comment	703478-102 PF05297909 for K. Henegar VT 100C			Date	21 04 2010 12 01 40
Date Stamp	21 Oct 2010 17	24:16		File Name	USEG7NMBD	ATAIHENEGARIN ZELLM D	21 001 2010 17:24:16
Frequency (MHz)	599.87	Nucleus	1H	Number of Transients	8	Origin	P05297909-210C12010/4/PDATA(1\1r
Original Points Count	32768	Owner	staff	Points Count	32768	Bulco Seguence	spect
Receiver Gain	362.00	SW(cyclical) (Hz)	10775.86	Solvent	DMSO de	Fuise sequence	2930
Spectrum Type	STANDARD	Sweep Width (Hz)	10775.53	Temperature (degree C	100,029	Spectrum Offset (Hz)	2228.5520





















Acquisition Time (sec)	3.9846	Comment	703478x172 desily	3478x172 desilylated PROTON CDCl3 /opt/topspin3.0/data kehenega					
Date	10 Aug 2011	10:15:28		Date Stamp	10 Aug 2011 10				
File Name	\SEG3\SAME	A2\BH010310\KEHENEGA\10	082011-KEHENEGA	3\PDATA\1\1r		Frequency (MHz)	400.13		
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768		
Owner	nmr	Points Count	85538	Pulse Sequence	zg30	Receiver Gain	57.18		
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.4238	Spectrum Type	STANDARD		
Sweep Width (Hz)	8223.56	Temperature (degree C) 22.308						





2/2/2012 11.48.47 AM





Acquisition Time (sec)	3.9846	Comment	705309x005 solid p	309x005 solid product PROTON CDCI3 /opt/topspin3.0/data kehenega						
Date	09 Aug 2011 1	2:29:52		Date Stamp	09 Aug 2011 12	2:29:52				
File Name	ISEG3ISAMB	A2\BH010310\KEHENEGA\090	082011-KEHENEGA	A\3\PDATA\1\1r Frequency (MHz)			400.13			
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768			
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	32.34			
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.4238	Spectrum Type	STANDARD			
Sweep Width (Hz)	8223.56	Temperature (degree C								





Acquisition Time (sec)	1.3631	Comment	705309x005 solid product 13C night CDCI3 /opt/topspin3.0/data kehenega					
Date	09 Aug 2011 1	3:53:04		Date Stamp	09 Aug 2011 13	:53:04		
File Name	ISEG3ISAMB	A2\BH010310\KEHENEGA\090	82011-KEHENEGA	PDATA\1\1r		Frequency (MHz)	100.61	
Nucleus	13C	Number of Transients	2048	Origin	spect	Original Points Count	32768	
Owner	nmr	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	184.55	
SW(cyclical) (Hz)	24038.46	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10055,4414	Spectrum Type	STANDARD	
Sween Width (Hz)	24037 73	Temperature (degree (1 24 138	NEWS CONTRACTOR OF CALIFORNIA -	04340404022	CLOSE CONCOURSES	2-804 000 E BAC 340	



6c Major Diastereomer



Acquisition Time (sec)	3.9846	Comment	705309x009 Low R	product PROTON CDCI3 /opt/topspin3.0/data kehenega					
Date	16 Aug 2011 08:	05:20		Date Stamp	16 Aug 2011 08	3:05:20			
File Name	\\SEG3\SAMBA2	BH010310\KEHENEGA\16	082011-KEHENEGA	1\PDATA\1\1r		Frequency (MHz)	400.13		
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768		
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	15.29		
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2470.9683	Spectrum Type	STANDARD		
Sweep Width (Hz)	8223.56	Temperature (degree (21.947		and the second second				
and the second state of the second	Charles and the second second second	Same Salar and Salar	262						



6c Major Diastereomer

6c Minor Diastereomer

Acquisition Time (sec)	3.9846	Comment	705309x008 minor	(solid) diastereomer PRO	TON CDCI3 /opt/to	opspin3.0/data kehenega		
Date	18 Aug 2011 09:	52:00		Date Stamp	18 Aug 2011 09	:52:00		
File Name	\\SEG3\SAMBA2	BH010310\KEHENEGA\180	82011-KEHENEGA	PDATA\1\1r		Frequency (MHz)	400.13	
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768	
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	93.74	
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2470.9683	Spectrum Type	STANDARD	
Sweep Width (Hz)	8223.56	Temperature (degree C	22.009					

6c Minor Diastereomer

Acquisition Time (sec)	1.3631	Comment	705309x009 low Rf	solids 13C night CDCI3 /	opt/topspin3.0/data	a kehenega		
Date	29 Aug 2011 16	3:52:16		Date Stamp	29 Aug 2011 16	52:16		
File Name	\\SEG3\SAMBA	2\BH010310\KEHENEGA\290	82011-KEHENEGA	2\PDATA\1\1r	36	Frequency (MHz)	100.61	
Nucleus	13C	Number of Transients	2048	Origin	spect	Original Points Count	32768	
Owner	nmr	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	184.55	
SW(cyclical) (Hz)	24038.46	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10056.9092	Spectrum Type	STANDARD	
Sweep Width (Hz)	24037.73	Temperature (degree C	24.050	7 //040 (A - 0.040 (A	~7 (XS-3862-0580-			

			1000102000 1110	IFROTON COCIS /Op/10ps	into:ordata keneneg	d Date	28 060 2011 10:20:40
Date Stamp	29 Sep 2011 16:	26:40	144.6	File Name	\\SEG3\SAMBA2\	BH010310\KEHENEGA\	29092011-KEHENEGA\4\PDATA\1\1r
Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	16	Origin	spect
Driginal Points Count	32768	Owner	nmr	Points Count	65536	Pulse Sequence	zg30
leceiver Gain	82.63	SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	2465.4238	Spectrum Type	STANDARD	Sweep Width (Hz)	8223.56	Temperature (degree	C) 25.004
0.40 0.35 0.30 0.25 0.20 0.15	ENEGA WEAR	StaleFactor = 4		4.136.	KK_K	e.70 e.30 e.e5 3	9.78 90.0145.00 30.74

	4.0830	Comment	proton 373K VT	for K. Henegar Possible rot	amers	Date	06 Oct 2011 11:28:00
Date Stamp	06 Oct 2011 11:	28:00		File Name	\\SEG7\NMRDAT	A\HENEGAR\N-ZELLM-H	ENEGAR-VT 60CT201114/PDATA\1\1
requency (MHz)	499.87	Nucleus	1H	Number of Transients	4	Origin	spect
Original Points Count	32768	Owner	omr	Points Count	65536	Pulse Sequence	zg30
Receiver Gain	31.36	SW(cyclical) (Hz)	6996.27	Solvent	DMSO-d6	Spectrum Offset (Hz)	2999.2207
pectrum Type	STANDARD	Sweep Width (Hz)	6996.16	Temperature (degree C) 100.108	6	
0.15				J	9.39.9.40		56-1-97 56-

7a OTMS

7a OTMS

OTMS

7d BnO

	OTMS
7d	BnO CCl ₃

8d BnO CCI3

Date

Acquisition Time (sec) 3,9846

Comment

03 Aug 2011 08:18:08

705309x0004 silylated PROTON CDCl3 /opt/topspin3.0/data kehenega Date Stamp 03 Aug 2011 08:18:08 82011-KEHENEGA\1\PDATA\1\1r Frequency (MHz) 400.13

File Name	ISEG3 SAMBA	2\BH010310\KEHENEGA\03	082011-KEHENEGA	1\PDATA\1\1r		Frequency (MHz)	400,13	
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768	
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	82.63	
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.1733	Spectrum Type	STANDARD	
Sween Width (Hz)	8223 56	Temperature (degree (1 25 003					

Acquisition Time (sec)	1.3631	Comment	705309x004 silylat	ed 13C day CDCl3 /opt/to	pspin3.0/data keh	enega	
Date	10 Aug 2011 1:	2:34:08		Date Stamp	10 Aug 2011 12	:34:08	
File Name	ISEG3 SAMBA	218H010310/KEHENEGA1100	82011-KEHENEGA	8\PDATA\1\1r	30	Frequency (MHz)	100.61
Nucleus	13C	Number of Transients	256	Origin	spect	Original Points Count	32768
Owner	nmr	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	184.55
SW(cyclical) (Hz)	24038.46	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10057,6406	Spectrum Type	STANDARD
Sweep Width (Hz)	24037.73	Temperature (degree C	23.674	- 1994 - A. S. A. D. B. B. A. S. A. B.	2010-2010-2010-2000		2

And a second second second second

	3.8840	Comment	705309x0004 desily	lated PROTON CDCI3 /o	pt/topspin3.0/data ke	ehenega	
Date	04 Aug 2011 12	:00:00		Date Stamp	04 Aug 2011 12:00	0:00	
File Name	ISEG3 SAMBA	2\BH010310\KEHENEGA\040	82011-KEHENEGA	2\PDATA\1\1r		Frequency (MHz)	400.13
lucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	32.34
W(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.2983	Spectrum Type	STANDARD
weep Width (Hz)	8223.56	Temperature (degree C) 22.491				
04082011-KEHE 1.0- 0.9- 0.8- 0.7- 0.6- 0.6- 0.4- 0.3- 0.2- 0.1- 0.2- 0.1- 0.2- 0.1- 0.2- 0.1- 0.2	NEGA.0026Hica	1988aleFactor = 1	2284 2001		9.75	12 52 52 52 52 52 52 52 52 52 52 52 52 52	

	UOF ON CALIFORNIA			Date Stamp	01 Aug 2011 12.	5	100.04
File Name	ISEG3ISAMBA2	BHU1U310/KEHENEGA/U40	SZUTT-KEHENEGA	Orisis	1000	Prequency (MHZ)	100.01
Owner	130	Paints Count	200	Drigin Pulsa Sasuanaa	spect	Pagainar Points Count	32/08
SWier	24020.48	Folms Count	32700	Fuise Sequence	20051 0400	Receiver Gall	104.00 CTANDADD
SW(Cyclical) (Hz)	24038.40	Solvent	CHLOROFORM-0	Spectrum Offset (Hz)	10051.0400	Spectrum Type	STANDARD
1.0 0.9 0.8 0.7 0.7 0.7 0.7 0.7 0.7 0.7 0.7		scaleFactor = 1			77.31 76.68		

4/4/4014 4.01.7111 W

OTMS CCl₃ **7h** _{CH₃O}

cquisition Time (sec)	3.9846	Comment	4-methoxy, silvl eth	er PROTON CDCI3 /opt/t	opspin3.0/data ke	shenega		
ate	15 Sep 2011 16	:26:40	-	Date Stamp	15 Sep 2011 16	3:26:40		
e Name	\\SEG3\SAMBA	2\BH010310\KEHENEGA\15	092011-KEHENEGA	1\PDATA\1\1r	2080 CTRACTORING	Frequency (MHz)	400.13	
cleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768	
/ner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	105.47	
(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2465.2988	Spectrum Type	STANDARD	
eep Width (Hz)	8223.56	Temperature (degree C	25.000					
0.94 0.84 0.74 0.64 0.54 0.44 0.34 0.24 0.14	Vertica	19.62		3	88	60.02	88.	05

OTMS CCl₃ **7h** CH₃O

dle	15 Sep 2011 19:	28:00		Date Stamp	15 Sep 2011 19:	28:00	
le Name	\\SEG3\SAMBA2	BH010310/KEHENEGA/150	92011-KEHENEGA	PDATA\1\1r		Frequency (MHz)	100.61
ucleus	13C	Number of Transients	2048	Origin	spect	Original Points Count	32768
wner	nmr	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	184.55
V(cyclical) (Hz)	24038.46	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	10057.6582	Spectrum Type	STANDARD
eep Width (Hz)	24037.73	Temperature (degree C) 25.002				
1.0- 1.0-		159.46			77.31 76.68		

ŎН `CCl₃ 8h _{CH30}

Acquisition Time (sec)	3.9846	Comment	012 chromed solid	Is PROTON CDCI3 /opt	/topspin3.0/data kehen	ega	
Date	22 Sep 2011 14	:18:40		Date Stamp	22 Sep 2011 14:1	8;40	
File Name	ISEG3ISAMBA	2\BH010310\KEHENEGA\22	092011-KEHENEGA	AV2\PDATA\1\1r		Frequency (MHz)	400.13
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	32768
Owner	nmr	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	150.37
SW(cyclical) (Hz)	8223.68	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2465.2983
Spectrum Type	STANDARD	Sweep Width (Hz)	8223.56	Temperature (degre	e C) 25.004		

ОH CCl₃ 8h CH₃O

