

# Synthesis and Microstructural Characterization of Poly(chlorotrifluoroethylene-*co*-vinylidene chloride) Copolymers

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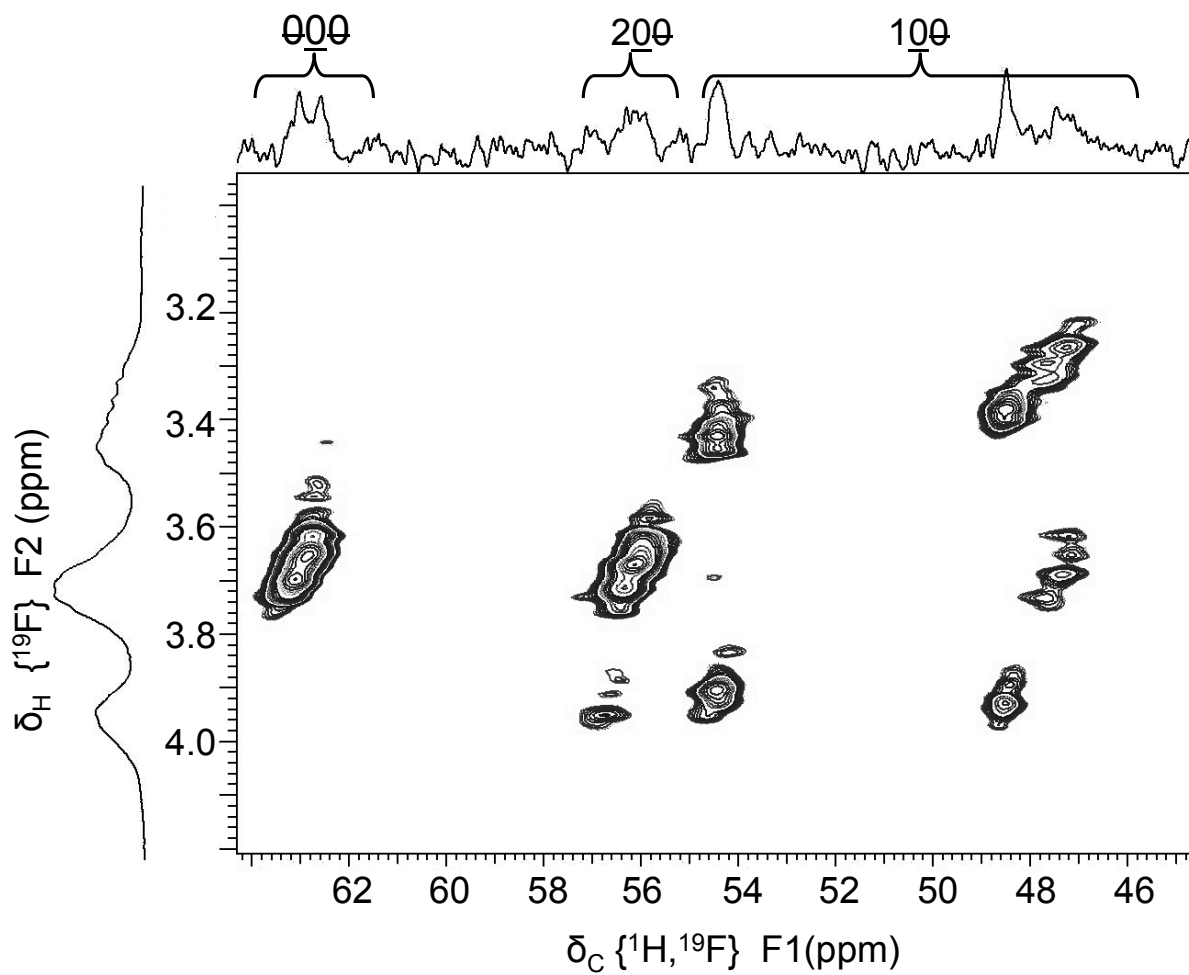
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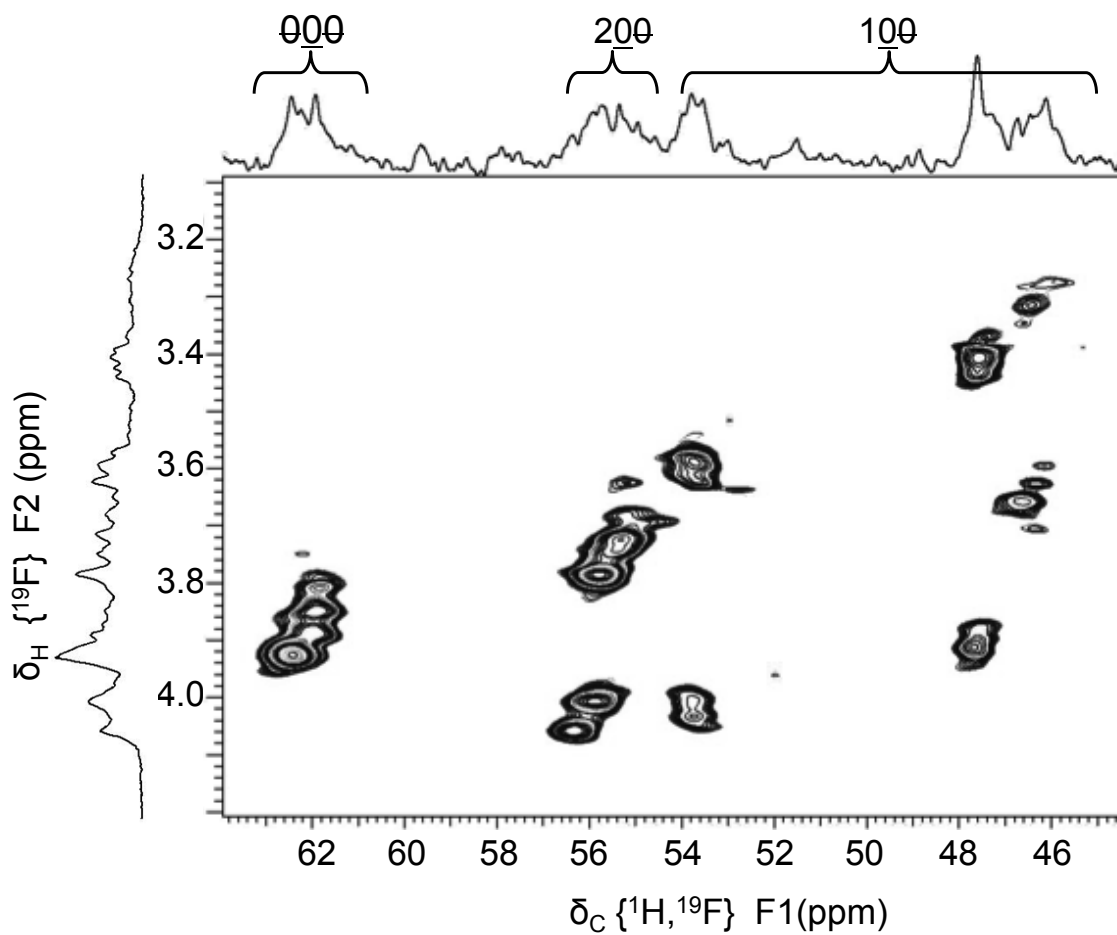
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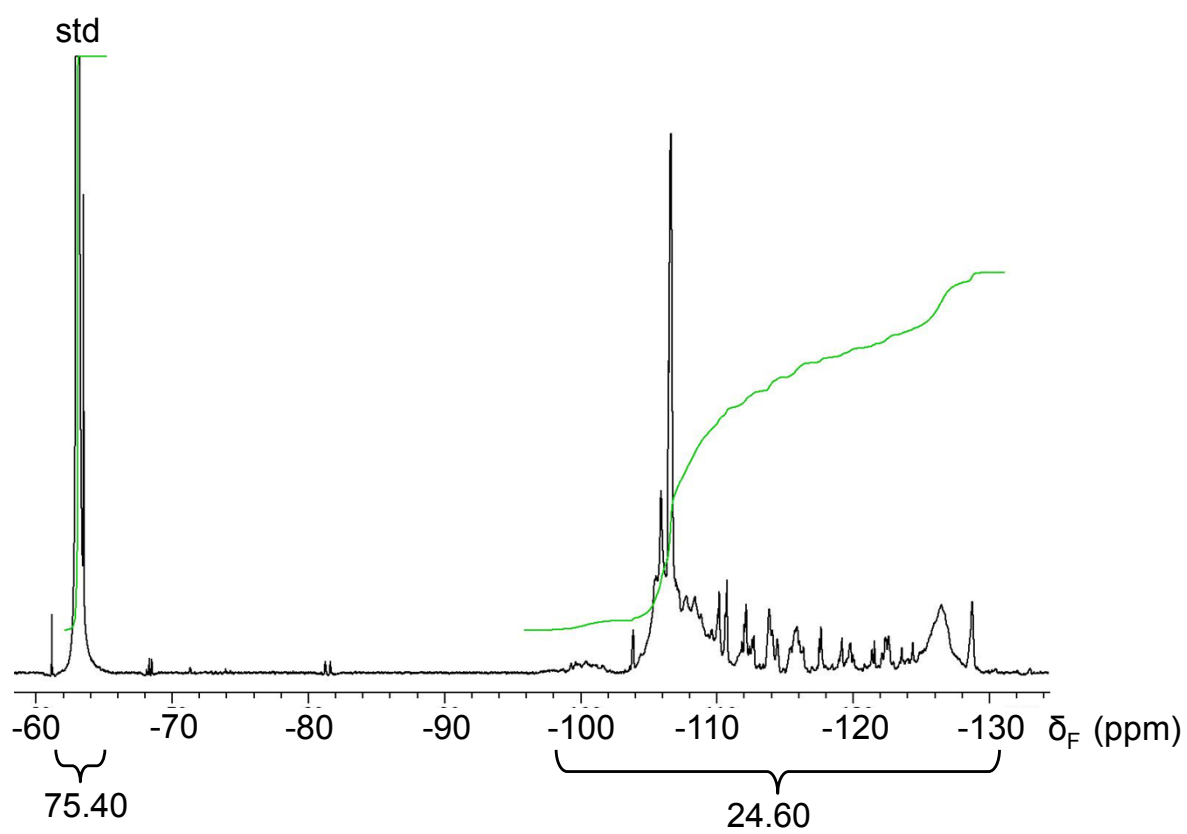
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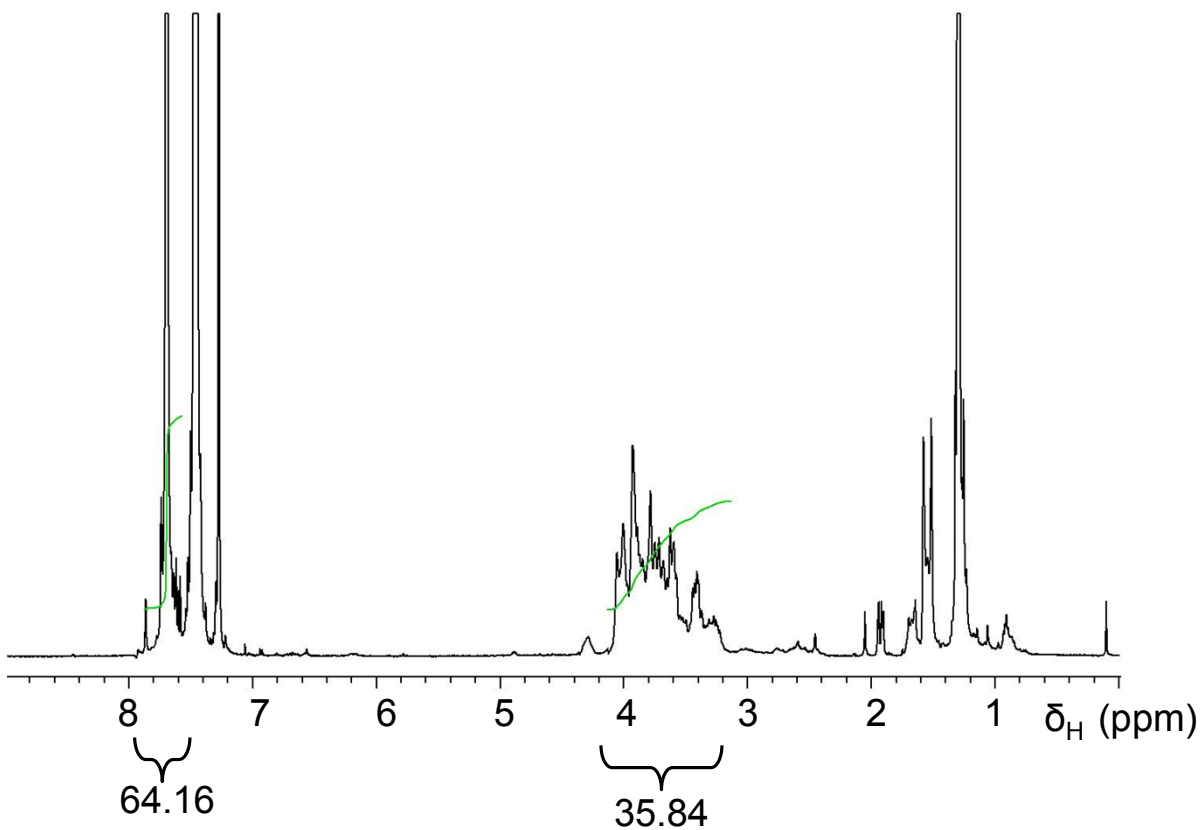
**Figure S1.** Selected region from the 500 MHz  ${}^1\text{H}\{^{13}\text{C}\}$  gHSQC spectrum of poly(CTFE-co-VDC) obtained in toluene- $d_8$  at 90 °C.



**Figure S2.** Selected region from the 500 MHz  $^1\text{H}\{^{13}\text{C}\}$  gHSQC spectrum of poly(CTFE-*co*-VDC) obtained in  $\text{CDCl}_3$  at 50  $^\circ\text{C}$ .



**Figure S3.** Quantitative 470 MHz  $^{19}\text{F}\{^1\text{H}\}$  1D-NMR spectrum of poly(CTFE-*co*-VDC) with internal standard in  $\text{CDCl}_3$  at 50  $^\circ\text{C}$ .



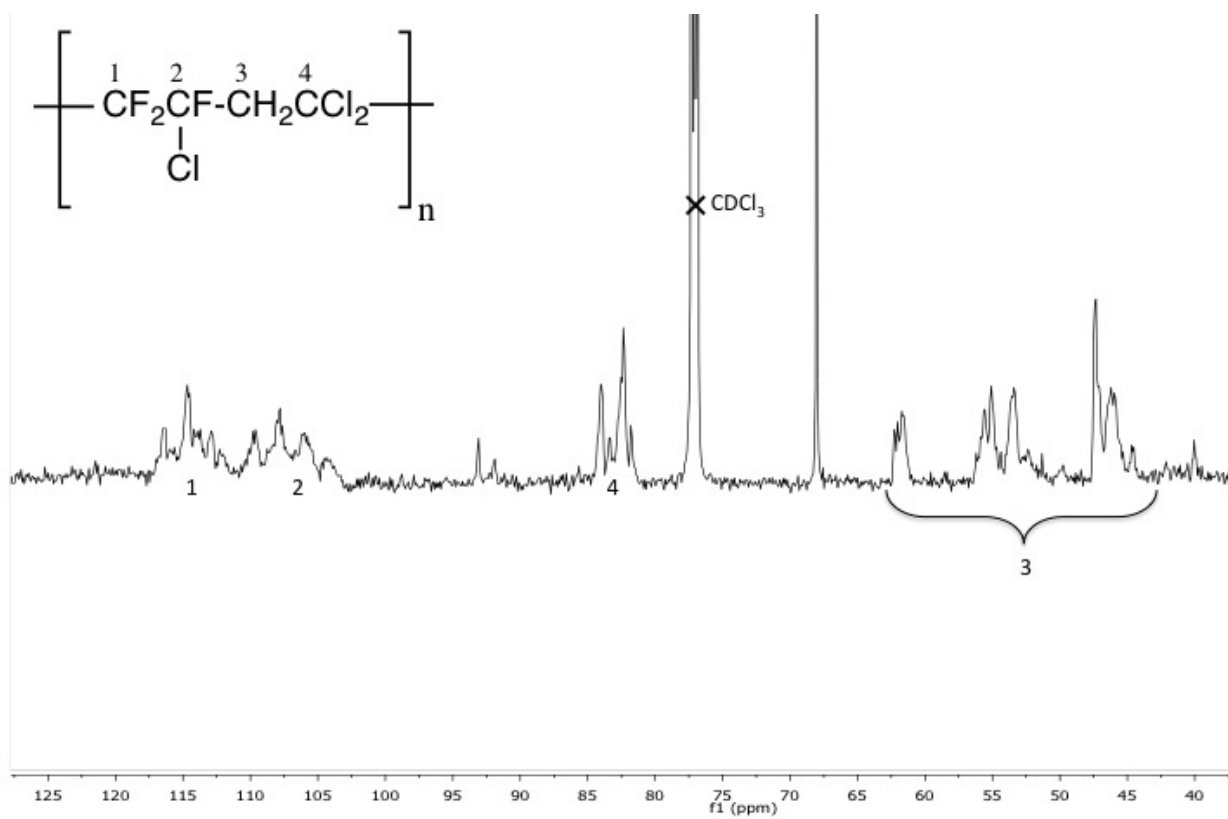
**Figure S4.** Quantitative 500 MHz  $^1\text{H}\{^{19}\text{F}\}$  1D-NMR spectrum of poly(CTFE-co-VDC) with internal standard in  $\text{CDCl}_3$  at 50  $^\circ\text{C}$ .

Determination of  $P_{\text{VDC}}$  from Fig. S3 and Fig. S4:

$$\frac{N_{\text{VDC}}}{N_{\text{std}}} = \frac{A_{\text{VDC}} / 2}{A_{\text{std}} / 1} = \frac{35.84 / 2}{64.16 / 1} = 0.28$$

$$\frac{N_{\text{CTFE}}}{N_{\text{std}}} = \frac{A_{\text{CTFE}} / 3}{A_{\text{std}} / 3} = \frac{24.60 / 3}{75.40 / 3} = 0.33$$

$$P_{\text{VDC}} = \frac{N_{\text{VDC}}}{N_{\text{VDC}} + N_{\text{CTFE}}} * 100\% = 46\%$$



**Figure S5.** Selected region from the 600 MHz  $^{13}\text{C}$  spectrum of poly(CTFE-co-VDC) obtained in  $\text{CDCl}_3$  at room temperature.

## Pulse Program Used for $^1\text{H}\{^{13}\text{C}\}$ HSQC Experiment

/\* gHSQC\_20141120 - Gradient Selected phase-sensitive HSQC

Features included:

F1 Axial Displacement

Parameters:

sspul : selects magnetization randomization option  
nullflg:selects TANGO-Gradient option  
hsglvl: Homospoil gradient level (DAC units)  
hsgt : Homospoil gradient time  
gzlvlE : encoding Gradient level  
gtE : encoding gradient time  
EDratio : Encode/Decode ratio  
gstab : recovery delay  
j1xh : One-bond XH coupling constant

KrishK- Last revision : June 1997

KrishK- Revised : July 2004

KrishK - Includes slp saturation option : July 2005

KrishK - includes purge option : Aug. 2006

\*\*\*\*v17,v18,v19 are reserved for PURGE \*\*\*

Linlin Li, Chun Gao and P Rinaldi - Added decoupler pulse on third channel in middle of evolution period Nov 2014 to standard gHSQC in version vnmrJ 3.2

\*/

#include <standard.h>

#include <chempack.h>

```
static int    ph1[4] = {1,1,3,3},
              ph2[2] = {0,2},
              ph3[8] = {0,0,0,0,2,2,2,2},
              ph4[16] = {0,0,0,0,0,0,0,0,2,2,2,2,2,2,2,2},
              ph5[16] = {1,3,3,1,3,1,1,3,3,1,1,3,1,3,3,1};
```

pulsesequance()

```
{
  double
    pwxlvl = getval("pwxlvl"),
    pwx2lvl = getval("pwx2lvl"),
    pwx = getval("pwx"),
    pwx2 = getval("pwx2"),
    gzlvlE = getval("gzlvlE"),
```

```

        gtE = getval("gtE"),
        EDratio = getval("EDratio"),
        gstab = getval("gstab"),
        mult = getval("mult"),
        hsglvl = getval("hsglvl"),
        hsgt = getval("hsgt"),
        tau,d2correction,
        taug;
int    icosel,
        prgcycle = (int)(getval("prgcycle")+0.5),
        phase1 = (int)(getval("phase")+0.5),
        ZZgsign;

tau = 1/(4*(getval("j1xh")));

if (mult > 0.5)
    taug = 2*tau;
else
    taug = gtE + gstab + 2*GRADIENT_DELAY;
ZZgsign=-1;
if (mult == 2) ZZgsign=1;
icosel = 1;

assign(ct,v17);
assign(zero,v18);
assign(zero,v19);

if (getflag("prgflg") && (satmode[0] == 'y') && (prgcycle > 1.5))
{
    hlv(ct,v17);
    mod2(ct,v18); dbl(v18,v18);
    if (prgcycle > 2.5)
    {
        hlv(v17,v17);
        hlv(ct,v19); mod2(v19,v19); dbl(v19,v19);
    }
}

settable(t1,4,ph1);
settable(t2,2,ph2);
settable(t3,8,ph3);
settable(t4,16,ph4);
settable(t5,16,ph5);

getelem(t1, v17, v1);
getelem(t3, v17, v3);

```



```

getelem(t4, v17, v4);
getelem(t2, v17, v2);
getelem(t5, v17, oph);

assign(zero,v6);
add(oph,v18,oph);
add(oph,v19,oph);

/*
  mod2(id2,v14);
  dbl(v14,v14);
*/
initval(2.0*(double)(((int)(d2*getval("sw1")+0.5)%2)),v14);

if ((phase1 == 2) || (phase1 == 5))
  icosel = -1;

add(v2,v14,v2);
add(oph,v14,oph);

status(A);
obspower(tpwr);
dec2power(pwx2lv1);
delay(5.0e-5);
if (getflag("sspul"))
  steadystate();

if (satmode[0] == 'y')
{
  if ((d1-satdly) > 0.02)
    delay(d1-satdly);
  else
    delay(0.02);
  if (getflag("slpsat"))
  {
    shaped_satpulse("relaxD",satdly,zero);
    if (getflag("prgflg"))
      shaped_purge(v6,zero,v18,v19);
  }
  else
  {
    satpulse(satdly,zero,rof1,rof1);
    if (getflag("prgflg"))
      purge(v6,zero,v18,v19);
  }
}
}

```

```

else
    delay(d1);

if (getflag("wet"))
    wet4(zero,one);

decpower(pwxlvl);

status(B);

if (getflag("nullflg"))
{
    rgpulse(0.5*pw,zero,rof1,rof1);
    delay(2*tau);
    simpulse(2.0*pw,2.0*pwx,zero,zero,rof1,rof1);
    delay(2*tau);
    rgpulse(1.5*pw,two,rof1,rof1);
    zgradpulse(hsglvl,hsgt);
    delay(1e-3);
}

rgpulse(pw,v6,rof1,rof1);
delay(tau);
simpulse(2*pw,2*pwx,zero,zero,rof1,rof1);
delay(tau);
rgpulse(pw,v1,rof1,rof1);
    zgradpulse(hsglvl,2*hsgt);
    delay(1e-3);
decrpulse(pwx,v2,rof1,2.0e-6);

    d2correction=pw;
    if (2.0*pwx2>pw) d2correction=2.0*pwx2;

if (d2/2 - d2correction - 6.0e-6 > 0.0)
    delay(d2/2 - d2correction - 6.0e-6);
else
    delay(d2/2);
dec2rgpulse(pwx2,zero, 2.0e-6,2.0e-6);
sim3pulse(2*pw,(double) 0.0,2*pwx2,zero,zero,one,2.0e-6,2.0e-6);
dec2rgpulse(pwx2,zero, 2.0e-6,2.0e-6);
if (d2/2 - d2correction - 6.0e-6 > 0.0)
    delay(d2/2 - d2correction - 6.0e-6);
else
    delay(d2/2);
zgradpulse(gzlvlE,gtE);
delay(taug - gtE - 2*GRADIENT_DELAY);

```

```

simpulse(mult*pw,2*pwx,zero,zero,rof1,rof1);
delay(taug - (2*pwx/PI) - 2.0e-6);

decrpulse(pwx,v4,2.0e-6,rof1);
    zgradpulse(ZZgsign*0.6*hsglvl,1.2*hsgt);
    delay(1e-3);
rgpulse(pw,v3,rof1,rof1);
delay(tau - (2*pw/PI) - 2*rof1);
simpulse(2*pw,2*pwx,zero,zero,rof1 , rof2);
decpower(dpwr);
dec2power(dpwr2);
zgradpulse(icosel*2.0*gzlvlE/EDratio,gtE/2.0);
delay(tau - gtE/2.0 - 2*GRADIENT_DELAY - POWER_DELAY);
status(C);
}

```