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COMPUTER ANALYSIS OF NON-ISOTHERMAL TG DATA FOR MECHANISM AND ACTIVATION ENERGY. PART I

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ABSTRACT

A computer program (CP) is presented, based on a non-computer procedure reported in the literature for the determination of mechanism and corresponding activation energy utilizing non-isothermal TG data. This CP is then applied to six sets of TG data. The agreement between estimated and reported results is excellent. Various aspects and limitations of the CP are discussed.

INTRODUCTION

The authors recently presented a computer program whereby one of twelve theoretically possible solid-state decomposition mechanisms could be distinguished utilizing non-isothermal (NI) or isothermal TG data [1]. Zsako [2] also presented a method for the kinetic analysis of NI TG data for mechanism (and for activation energy, E). However, this author did not employ a computer procedure. Instead, Tables were used which consisted of so-called $-\log p(x)$ values corresponding to different temperatures and E-values. Further, analysis of NI TG data was confined to only three possible mechanisms, i.e., $n = 0, 1, 2$, assuming an n-order type reaction. In this paper, the authors intend to present a computer program based on the procedure outlined by Zsako and, by applying this program to NI TG data, to distinguish one of ten theoretically possible solid-state decomposition mechanisms and to determine the corresponding value of E.

THEORY

Prior to presenting and discussing the aforementioned computer program, some theoretical background would appear to be apropos.

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It has been previously indicated [3] that

$$g(\alpha) = ZEp(x)/Rq \tag{1}$$

where $g(\alpha) = \int_0^\alpha d\alpha/f(\alpha)$; Z = pre-exponential factor; R = gas constant; q = constant heating rate; and, $p(x) = e^{-x}/x - \int_x^\infty e^{-u}du/u$, where $x = E/RT$. When the common logarithm of eqn. (1) is taken, the result is

$$B = \log(ZE/Rq) = \log g(\alpha) - \log p(x) \tag{2}$$

where B = constant. Further, using a truncated Schlömilch expansion [4]

$$p(x) = e^{-x}(1/x)[1/(x+2)] \tag{3}$$

Doyle [5] has indicated that eqn. (3) yields an excellent approximation for the true value of $p(x)$ with values of x between 10 and 50. (We have found that eqn. (3) is still a good approximation up to values of $x = 60$). Since values of x are approximately equal to values of E (kcal mol^{-1}), E-values employed in the computer program were in the range 20–60 kcal mol^{-1} (cf. lines 35, 55, 75 and 145 of the BASIC computer program (BCPA) in Appendix).

THE COMPUTER PROGRAM

Before making a run, the value of N (number of alpha-T(K) data pairs) in line 15 should be adjusted, if necessary. Also, the following ten theoretical mechanisms were examined to ascertain which one of them conformed best to the NI TG data: A4, A3, and A2 (random nucleation, Avrami equations); R2 and R3 (phase boundary reaction, cylindrical symmetry and spherical symmetry, respectively); F1 (random nucleation, one nucleus per particle); D1, D2, D3, and D4 (corresponding to 1-dimensional diffusion, 2-dimensional diffusion–cylindrical symmetry, 3-dimensional diffusion–Jander spherical symmetry, and 3-dimensional diffusion–Ginstling/Brounshtein spherical symmetry). The mechanisms are further defined in lines 250 and 255 of the BCPA. In order to reduce to reasonable values the host of computations involved and the corresponding relatively long computer running time (to ca., 5–15 min), only ten arbitrarily chosen mechanisms were tested and values of E were incremented by only 1 kcal mol^{-1}.

Values of E ranging from 20–60 kcal mol^{-1} were assigned to each of the preceding mechanisms. Further, values of B (see eqn. (2)) were calculated for each pair of alpha-T(K) TG data at any particular E-value. An average value, $\bar{B}$, was then determined (line 90 of BCPA) and finally the standard deviation of B for each mechanism, denoted as Delta, for any particular E-value. Delta is defined as usual

$$\text{Delta} = \left[(B_i - \bar{B})^2/N\right]^{1/2} \tag{4}$$

(see lines 15, 95 of BCPA).

In the Appendix, a run is depicted which uses data from line 240. From this run, it can be seen that at $E = 20$ kcal mol^{-1}, the probable mechanism, based on the lowest value of Delta, was F1. When the E-value was increased to 21, the probable mechanism changed to D1 since its Delta now possessed the lowest value. These lowest values of Delta, and the corresponding values of E and the mechanism description (lines 250, 255) were stored for final computer analysis (lines 95, 135).

The computer run could be terminated in one of two ways. Thus, if the value of E attained 60 kcal mol^{-1}, all the lowest Delta-values for each E-value from 20–60 were listed along with their corresponding probable mechanisms. Then the lowest of these Delta-values was selected along with the corresponding E-value and mechanism and these final results were displayed (e.g., see last line of run in Appendix). However, in this type of termination, the printout will also indicate that the computer analysis was incomplete since the run was arbitrarily halted at $E = 60$ (line 145). Under these conditions, the results are uncertain and bear further investigation by other procedures.

The second manner in which the run may be terminated is as follows. From the run in the Appendix, it can be observed that, for the order in which the mechanisms are listed, as the E-value increases, the Delta-values which are physically above the Delta of the probable mechanism (DPM) increase, whereas the Delta-values which are physically below the DPM decrease. The value of the DPM may either increase or decrease with increasing E-value. Further, the magnitude of the Delta-values physically above the DPM increase as the physical position away from the DPM increases. The same holds for the magnitude of the Delta-value physically below the DPM. From the preceding, when the DPM of the last mechanism, D3, begins to increase, all the previously listed mechanisms have been tested, and the run simply ends (lines 140, 155–165, 170).

RESULTS AND DISCUSSION

The TG data presented in lines 185–240 of the BCPA were analyzed by the previously-described computer method. In the following are given in order, the line number containing the analyzed data, the computer-calculated and the reported E-values (kcal mol^{-1}), the computer-determined and the reported mechanism, whether or not the analysis was complete (Y/N), and finally, the reference from which the data was obtained: 185, 29, 28.9, F1, F1, Y, [2]; 195, 26, 28.3, R3, F1, Y, [2]; 205, 22, 22, R3, $n = 0.7$, Y, [6]; 215–220, 30, 30, D2, D2, Y, [7]; 230, 28, 28, R2, R2, N, [6]; 240, 30, 30, D3, D3, Y, [8]. From the preceding, it can be seen that the agreement between calculated and reported results is excellent, except for the data used in line 195. Thus, Zsako tested only 3 n-order type mechanisms and determined that the data best fitted $n = 1$ (F1). However, the computer procedure yielded an R3-mechanism and a corresponding $E = 26$. These results were checked using another computer procedure previously reported [9]. This procedure afforded values of $n = 0.64$ (equivalent to R3) and $E = 25$ kcal mol^{-1}. Also, all analyses were complete except for the analysis of data in line 230.

Some limitations of the program are: *E*-values estimated are at best accurate to ±1 kcal mol^{-1}; only 10 mechanisms were tested; and, an incomplete analysis can result. In order to lessen the running time, *E*-values were incremented by 1 and only 10 mechanisms were tested. If time is not an important consideration, value of *E* may be incremented by less than 1 kcal mol^{-1} and the number of mechanisms to be tested can be increased. However, in the latter case, in order to maintain the manner in which the DPM changes as previously described, additional mechanisms should be placed in a certain order [10]. Thus, P3 $[\mathrm{A}_{\wedge}(1/2)]$ should be placed between A3 and A2, and A1.5 $[-\log(1-\mathrm{A})_{\wedge}(2/3)]$ between A2 and R2, etc. Obviously, the run-time is also dependent on the value of *N*.

Whether or not an incomplete computer analysis results depends, in part, on the true *E*-value and also on the initial physical location of the DPM. Thus, from the order of the mechanisms listed in the run in the Appendix, it can be seen that if the initial DPM is at A4, then a large increase in *E* might be anticipated in order to reach D3. This increase could reach $E = 60$ and thereby cause an incomplete analysis to result. However, when the initial DPM starts at F1 (as in the run in the Appendix), there is less likelihood that $E = 60$ would be attained.

APPENDIX

A BASIC computer program for analysis of non-isothermal TG data for mechanism and activation energy.

```
10  TEXT : HOME
15 N = 11: REM  NO. DATA PRS.
20 Q = 1:FF = 1:EI = 20000
25  DIM ST$(50),SS$(50),DL(50),W(N),T(N),B(N),TS$(50),LD(50),LS(50),AE(50)

30 R = 1.9B7:F = 2.303:DL(0) = 100:LD(0) = 100:LS(0) = 100:EE = 0
35  DEF  FN P(X) = (X -  LOG ((1 / X) * (1 / (X + 2)))) / F: REM   SCHLÖMI
     LCH APPROXMN.
37 :
40  FOR J = 1 TO N: READ W(J),T(J): NEXT J
45  FOR J = 1 TO 10: READ ST$(J):SS$(J) = ST$(J): NEXT J
50 :
55 E = Q: REM   EA=20-60 KCAL/MOL
57 :
60  FOR CC = 1 TO 10
65  ON CC GOSUB 270,280,300,320,330,340,350,360,370,380
67 :
70  FOR K = 1 TO N
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75 EA = (EI + 1000 * (FF * E - 1)):X = EA / (R * T(K))
80 B(K) =  FN W(W(K)) +  FN P(X)
85  NEXT K
87 :
90  FOR L = 1 TO N:BA = BA + B(L): NEXT L:BA = BA / N
95  FOR M = 1 TO N:DI = DI + (B(M) - BA) ^ 2: NEXT M:DL(CC) =  SQR (DI / N
    ) + DL(CC)
100 DI = 0:BA = 0
105  NEXT CC
110 EE = EE + 1:LS(EE) = DL(CC - 1)
112 :
115  FOR I = 1 TO CC - 1
120  PRINT "FOR "ST$(I);", DELTA= "; INT (DL(I) * 1E4 + .5) / 1E4
125  IF DL(I) <  = DL(I - 1) THEN 135
130 DL(I) = DL(I - 1):ST$(I) = ST$(I - 1)
135  NEXT I: PRINT : PRINT "PROB.MECHNSM.= ";ST$(I - 1)" FOR EA= ";EA / 10
     00;" K/M":LD(Q) = DL(I - 1):TS$(Q) = ST$(I - 1):AE(Q) = EA: FOR J = 1
      TO 12:DL(J) = 0: NEXT J: PRINT
140  IF LS(EE) > LS(EE - 1) THEN 155
145 Q = Q + 1: IF Q = 42 THEN  FOR J = 1 TO 41: PRINT  INT (LD(J) * 1E4 +
     .5) / 1E4;"---";TS$(J): NEXT J: PRINT : PRINT "ANALYSIS INCOMPLETE!!;
      RESULTS OBTAINED ARE: ": GOTO 155
150  FOR K = 1 TO 10:ST$(K) = SS$(K): NEXT K: GOTO 55
155  FOR J = 1 TO EE: IF LD(J) <  = LD(J - 1) THEN 165
160 LD(J) = LD(J - 1):TS$(J) = TS$(J - 1):AE(J) = AE(J - 1)
165  NEXT J
167 :
170  PRINT : PRINT "EA= "AE(J - 1) / 1000" KCAL/MOL FOR MECHNSM. ";TS$(J -
     1);", DELTA= ";LD(J - 1)
175 :
180  END
182 :
185  REM DATA.03398,433.2,.10194,443.2,.19903,453.2,.36893,463.2,.58738,47
     3.2,.86893,483.2,.91748,493.2: REM CMPLX.I, ZSAKO DATA-->EA=29K/M, F1
     -MECH. (7 PRS.)
190 :
195  REM DATA .011236,413.2,.022472,423.2,.039326,433.2,.078652,443.2,.191
     01,453.2,.34270,463.2,.58427,473.2,.87640,483.2,.97191,493.2: REM   C
     MPLX. II, ZSAKO DATA-->EA=26K/M; R3-MECHNSM. (9-PRS.)
200 :
205  REM DATA.208,408.2,.3,413.2,.403,418.2,.528,423.2,.667,428.2,.806,433
     .2,.917,438.2: REM  REICH, NAHCO3, TA,24,9(1978): DATA-->EA=22 FOR R3
     -MECHNSM. (7-PRS.)
210 :
215  REM  DATA.04438,602,.0577,614,.08392,632,.1194,650,.16635,668,.22714,
     686,.2765,698,.3336,710,.39897,722,.4348,728,.4728,734,.5128,740,.554
     8,746,.5988,752: REM  DATA CONT'D. ON LN #220
217 :
220  REM  DATA.6444,758,.6916,764,.73986,770,.78884,776,.8379,782,.8861,78
     8,.9321,794,.9735,800: REM  HEIDE DATA-->30K/M FOR D2-MECHNSM. (22-PR
     S.)
225 :
230  REM DATA.1319,405,.20195,410,.30261,415,.44105,420,.61869,425,.81878,
     430,.97883,435: REM    THEOR.DATA, REICH, EA-->28.0 K/M, R2-MECHNSM.
235 :
240  DATA  .07197,620,.10695,640,.15439,660,.21661,680,.29533,700,.39099,7
     20,.50186,740,.62308,760,.74587,780,.85746,800,.94279,820: REM   REIC
     H,TA,34,287(1979)(D3-MECH);DATA-->30K/M FOR D3-MECHNSM. (11 PRS.)
245 :
250  DATA   "A4: (-LN(1-A))^(1/4)","A3: (-LN(1-A))^(1/3)","A2: (-LN(1-A))^
     (1/2)","R2: 1-(1-A)^(1/2)","R3: 1-(1-A)^(1/3)","F1: -LN(1-A)"
255  DATA    "D1: A^2","D2: A+(1-A)LN(1-A)","D4: 1-(2A/3)-(1-A)^(2/3)","D3
     : (1-(1-A)^(1/3))^2"
260 :
270  DEF  FN W(X) =  LOG (( -  LOG (1 - X)) ^ (1 / 4)) / F: RETURN
280  DEF  FN W(X) =  LOG (( -  LOG (1 - X)) ^ (1 / 3)) / F: RETURN
300  DEF  FN W(X) =  LOG (( -  LOG (1 - X)) ^ (1 / 2)) / F: RETURN
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```
320  DEF  FN W(X) =  LOG (1 - (1 - X) ^ (1 / 2)) / F: RETURN
330  DEF  FN W(X) =  LOG (1 - (1 - X) ^ (1 / 3)) / F: RETURN
340  DEF  FN W(X) =  LOG ( -  LOG (1 - X)) / F: RETURN
350  DEF  FN W(X) =  LOG (X ^ 2) / F: RETURN
360  DEF  FN W(X) =  LOG (X + (1 - X) *  LOG (1 - X)) / F: RETURN
370  DEF  FN W(X) =  LOG (1 - (2 * X / 3) - (1 - X) ^ (2 / 3)) / F: RETURN

380  DEF  FN W(X) =  LOG ((1 - (1 - X) ^ (1 / 3)) ^ 2) / F: RETURN
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]RUN
FOR A4: (-LN(1-A))^(1/4), DELTA= .4915
FOR A3: (-LN(1-A))^(1/3), DELTA= .4506
FOR A2: (-LN(1-A))^(1/2), DELTA= .3689
FOR R2: 1-(1-A)^(1/2), DELTA= .1962
FOR R3: 1-(1-A)^(1/3), DELTA= .1733
FOR F1: -LN(1-A), DELTA= .1259
FOR D1: A^2, DELTA= .1285
FOR D2: A+(1-A)LN(1-A), DELTA= .1819
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .2084
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .2675

PROB.MECHNSM.= F1: -LN(1-A) FOR EA= 20 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .5188
FOR A3: (-LN(1-A))^(1/3), DELTA= .4779
FOR A2: (-LN(1-A))^(1/2), DELTA= .3962
FOR R2: 1-(1-A)^(1/2), DELTA= .2235
FOR R3: 1-(1-A)^(1/3), DELTA= .2006
FOR F1: -LN(1-A), DELTA= .1528
FOR D1: A^2, DELTA= .107
FOR D2: A+(1-A)LN(1-A), DELTA= .1556
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .1814
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .2402

PROB.MECHNSM.= D1: A^2 FOR EA= 21 K/M

FOR A4: (-LN(1-A))^(1/4). DELTA= .5461
FOR A3: (-LN(1-A))^(1/3), DELTA= .5052
FOR A2: (-LN(1-A))^(1/2), DELTA= .4235
FOR R2: 1-(1-A)^(1/2), DELTA= .2507
FOR R3: 1-(1-A)^(1/3), DELTA= .2279
FOR F1: -LN(1-A). DELTA= .1797
FOR D1: A^2, DELTA= .0888
FOR D2: A+(1-A)LN(1-A), DELTA= .1297
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .1546
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .2129

PROB.MECHNSM.= D1: A^2 FOR EA= 22 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .5734
FOR A3: (-LN(1-A))^(1/3), DELTA= .5325
FOR A2: (-LN(1-A))^(1/2), DELTA= .4508
FOR R2: 1-(1-A)^(1/2), DELTA= .278
FOR R3: 1-(1-A)^(1/3), DELTA= .2552
FOR F1: -LN(1-A), DELTA= .2068
FOR D1: A^2, DELTA= .0763
FOR D2: A+(1-A)LN(1-A), DELTA= .1046
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .1279
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .1856

PROB.MECHNSM.= D1: A^2 FOR EA= 23 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .6007
FOR A3: (-LN(1-A))^(1/3), DELTA= .5598
FOR A2: (-LN(1-A))^(1/2), DELTA= .4781
FOR R2: 1-(1-A)^(1/2), DELTA= .3052
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FOR R3: 1-(1-A)^(1/3), DELTA= .2825
FOR F1: -LN(1-A), DELTA= .2339
FOR D1: A^2, DELTA= .0723
FOR D2: A+(1-A)LN(1-A), DELTA= .0808
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .1017
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .1584

PROB.MECHNSM.= D1: A^2 FOR EA= 24 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .6279
FOR A3: (-LN(1-A))^(1/3), DELTA= .587
FOR A2: (-LN(1-A))^(1/2), DELTA= .5053
FOR R2: 1-(1-A)^(1/2), DELTA= .3325
FOR R3: 1-(1-A)^(1/3), DELTA= .3098
FOR F1: -LN(1-A), DELTA= .261
FOR D1: A^2, DELTA= .0783
FOR D2: A+(1-A)LN(1-A), DELTA= .0602
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .0761
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .1311

PROB.MECHNSM.= D2: A+(1-A)LN(1-A) FOR EA= 25 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .6552
FOR A3: (-LN(1-A))^(1/3), DELTA= .6143
FOR A2: (-LN(1-A))^(1/2), DELTA= .5325
FOR R2: 1-(1-A)^(1/2), DELTA= .3597
FOR R3: 1-(1-A)^(1/3), DELTA= .337
FOR F1: -LN(1-A), DELTA= .2881
FOR D1: A^2, DELTA= .0923
FOR D2: A+(1-A)LN(1-A), DELTA= .0469
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .0524
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .1039

PROB.MECHNSM.= D2: A+(1-A)LN(1-A) FOR EA= 26 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .6824
FOR A3: (-LN(1-A))^(1/3), DELTA= .6415
FOR A2: (-LN(1-A))^(1/2), DELTA= .5598
FOR R2: 1-(1-A)^(1/2), DELTA= .3869
FOR R3: 1-(1-A)^(1/3), DELTA= .3643
FOR F1: -LN(1-A), DELTA= .3153
FOR D1: A^2, DELTA= .1113
FOR D2: A+(1-A)LN(1-A), DELTA= .0475
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .0343
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .0766

PROB.MECHNSM.= D4: 1-(2A/3)-(1-A)^(2/3) FOR EA= 27 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .7096
FOR A3: (-LN(1-A))^(1/3), DELTA= .6688
FOR A2: (-LN(1-A))^(1/2), DELTA= .587
FOR R2: 1-(1-A)^(1/2), DELTA= .4141
FOR R3: 1-(1-A)^(1/3), DELTA= .3915
FOR F1: -LN(1-A), DELTA= .3424
FOR D1: A^2, DELTA= .1332
FOR D2: A+(1-A)LN(1-A), DELTA= .0617
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .033
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .0494

PROB.MECHNSM.= D4: 1-(2A/3)-(1-A)^(2/3) FOR EA= 28 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .7369
FOR A3: (-LN(1-A))^(1/3), DELTA= .696
FOR A2: (-LN(1-A))^(1/2), DELTA= .6142
FOR R2: 1-(1-A)^(1/2), DELTA= .4414
FOR R3: 1-(1-A)^(1/3), DELTA= .4187
FOR F1: -LN(1-A), DELTA= .3696
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FOR D1: A^2, DELTA= .1568
FOR D2: A+(1-A)LN(1-A), DELTA= .0826
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .0499
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .0222

PROB.MECHNSM.= D3: (1-(1-A)^(1/3))^2 FOR EA= 29 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .7641
FOR A3: (-LN(1-A))^(1/3), DELTA= .7232
FOR A2: (-LN(1-A))^(1/2), DELTA= .6414
FOR R2: 1-(1-A)^(1/2), DELTA= .4686
FOR R3: 1-(1-A)^(1/3), DELTA= .4459
FOR F1: -LN(1-A), DELTA= .3967
FOR D1: A^2, DELTA= .1814
FOR D2: A+(1-A)LN(1-A), DELTA= .1065
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .0733
FOR D3: (1-(1-A)^(1/3))^2, DELTA= 5.1E-03

PROB.MECHNSM.= D3: (1-(1-A)^(1/3))^2 FOR EA= 30 K/M

FOR A4: (-LN(1-A))^(1/4), DELTA= .7913
FOR A3: (-LN(1-A))^(1/3), DELTA= .7504
FOR A2: (-LN(1-A))^(1/2), DELTA= .6687
FOR R2: 1-(1-A)^(1/2), DELTA= .4958
FOR R3: 1-(1-A)^(1/3), DELTA= .4731
FOR F1: -LN(1-A), DELTA= .4239
FOR D1: A^2, DELTA= .2066
FOR D2: A+(1-A)LN(1-A), DELTA= .1316
FOR D4: 1-(2A/3)-(1-A)^(2/3), DELTA= .0986
FOR D3: (1-(1-A)^(1/3))^2, DELTA= .0323

PROB.MECHNSM.= D3: (1-(1-A)^(1/3))^2 FOR EA= 31 K/M


EA= 30 KCAL/MOL FOR MECHNSM. D3: (1-(1-A)^(1/3))^2, DELTA= 5.06464008E-03

]
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