Self-Thickening Materials derived from Phenylpropanoid Ene Reactions

Atanu Biswas [1,*], H. N. Cheng [2,*], Bret Chisholm [1], Ryan Beni [1,3], Zengshe Liu [1], Karl Vermillion [1], Michael Appell [1], Kelton Forson [1], Omar El Seoud [4], Carlucio R. Alves [5], Roselayne F. Furtado [6]

[1] National Center for Agricultural Utilization Research, USDA Agricultural Research Services, 1815 N. University Street, Peoria, Illinois 61604, United States
[2] Southern Regional Research Center, USDA Agricultural Research Service, 1100 Allen Toussaint Boulevard, New Orleans, Louisiana 70124, United States
[3] Department of Chemistry, Tennessee State University, Nashville, Tennessee 37209, USA
[4] Institute of Chemistry, University of São Paulo, 05508-000 São Paulo, SP, Brazil
[5] State University of Ceará, Chemistry Department, Silas Munguba Av. 1.700, 60740-020, Fortaleza, CE, Brazil
[6] Embrapa Agroindústria Tropical, Rua Dra. Sara Mesquita 2270, CEP 60511-110 Fortaleza, CE, Brazil

* Correspondence: atanu.biswas@usda.gov (A.B.); hncheng100@gmail.com (H.N.C.)

Supplementary Materials

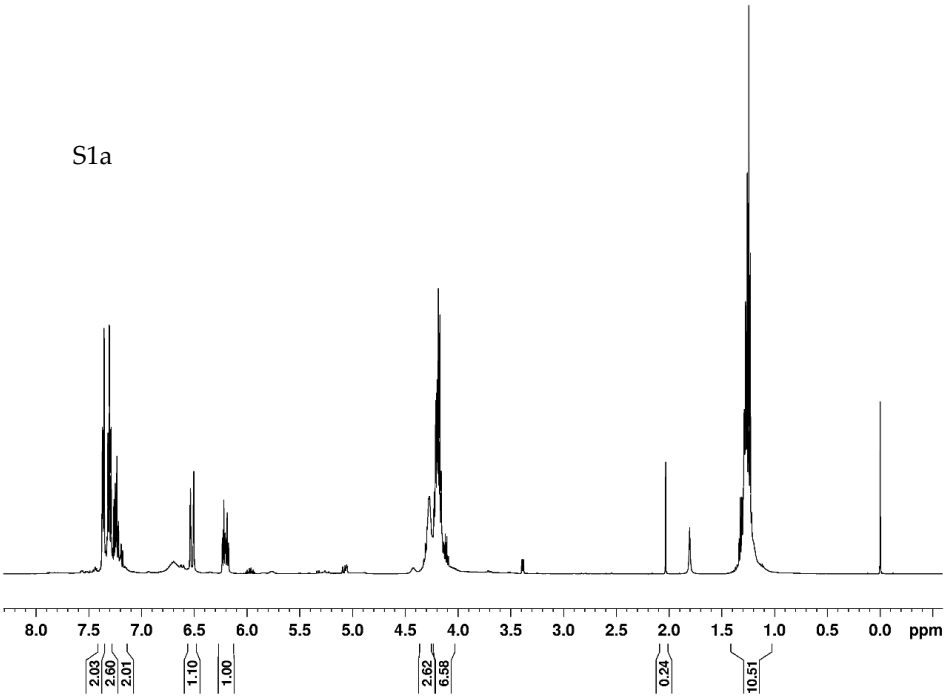
Figure S1. NMR spectra of the ene reaction products between allylbenzene and DEAD (sample A-2): a) ^{1}H spectrum with integration; b) 2D COSY plot (^{1}H-^{1}H shift correlation); c) 2D HSQC (^{1}H-^{13}C shift correlation).

Figure S2. NMR spectra of the purified ene reaction product between allylbenzene and DEAD: a) ^{13}C spectrum (upper plot), b) ^{1}H spectrum (lower plot). The letter A denotes the ene product and the subscripts correspond to the numbering shown in Scheme 3. The letter X indicates the $CDCl_3$ peaks.

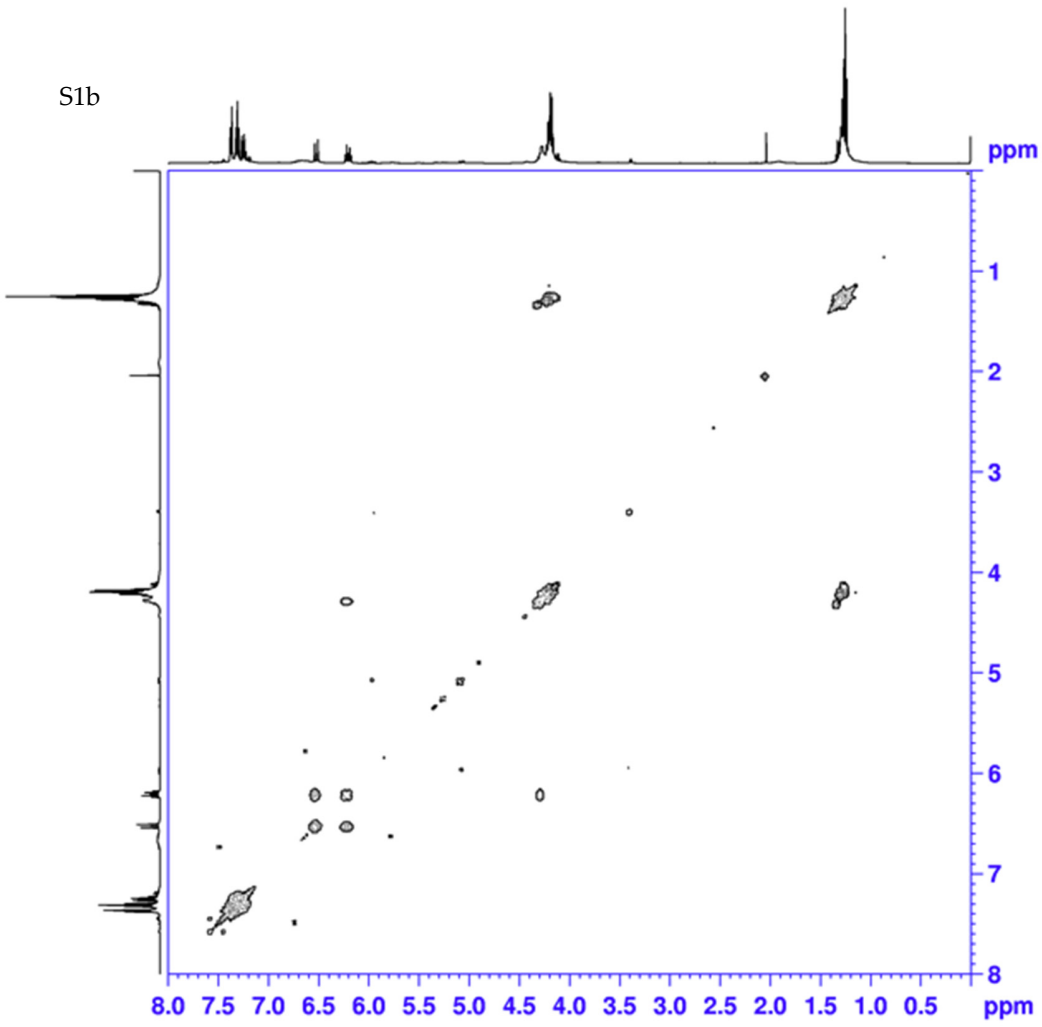
Figure S3. NMR spectra of the ene reaction products between methyl eugenol and DEAD (sample M-1): a) ^{1}H spectrum with integration; b) 2D COSY plot (^{1}H-^{1}H shift correlation); c) 2D HSQC (^{1}H-^{13}C shift correlation).

Figure S4. ^{1}H NMR spectrum (with integration) of the reaction products between eugenol and DEAD (sample E-1).

S1a


8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.03
2.60
2.01
1.10
1.00
2.62
6.58
0.24
10.51

S1b


ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm

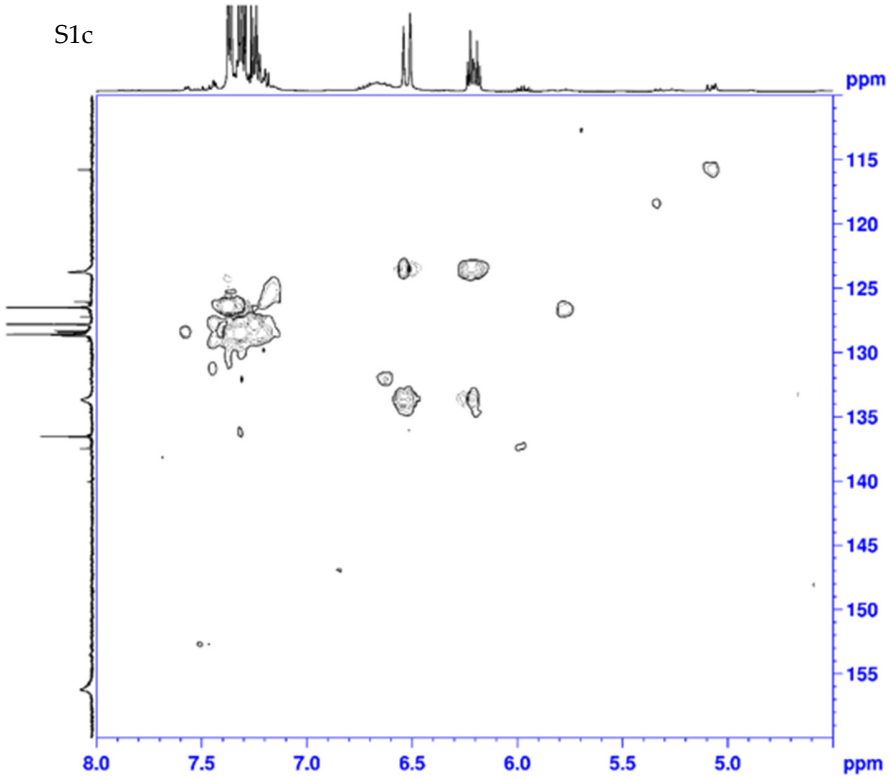


Figure S1. NMR spectra of the ene reaction products between allylbenzene and DEAD (sample A-2): a) ^{1}H spectrum with integration; b) 2D COSY plot (^{1}H-^{1}H shift correlation); c) 2D HSQC (^{1}H-^{13}C shift correlation).

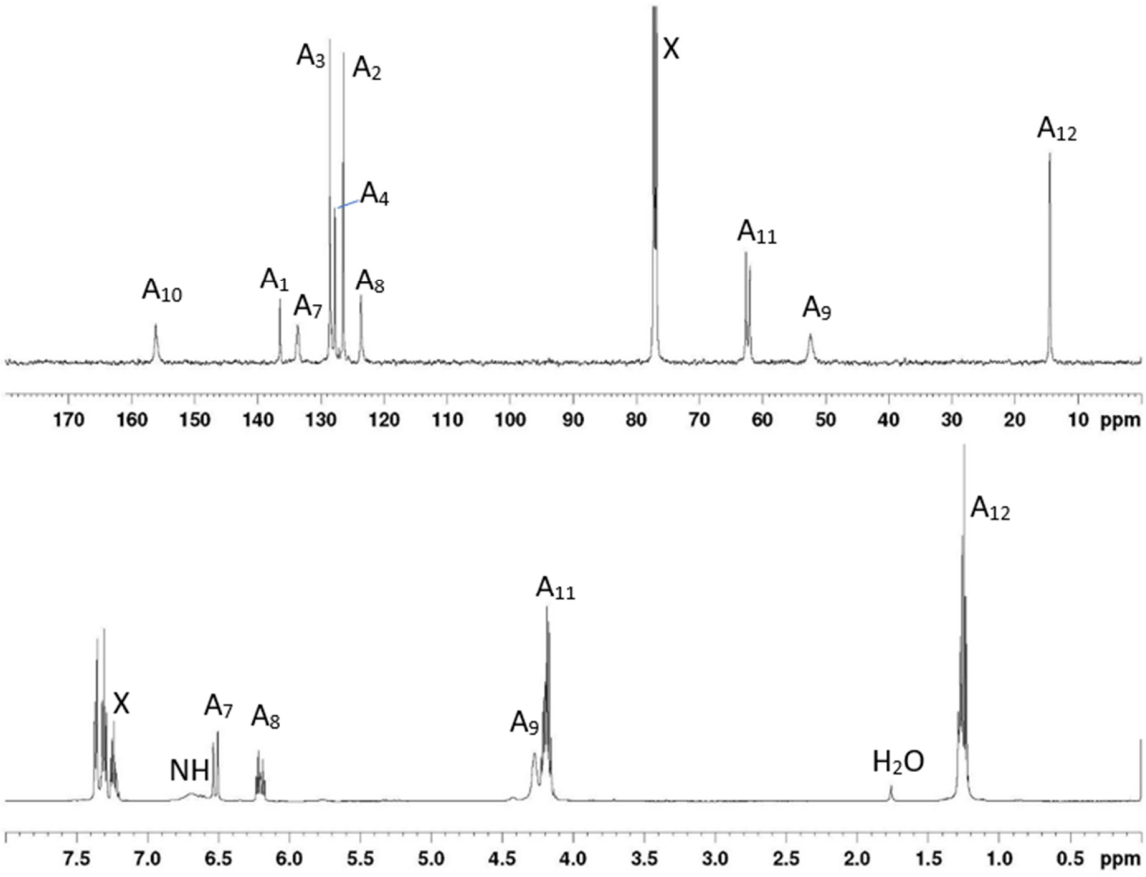
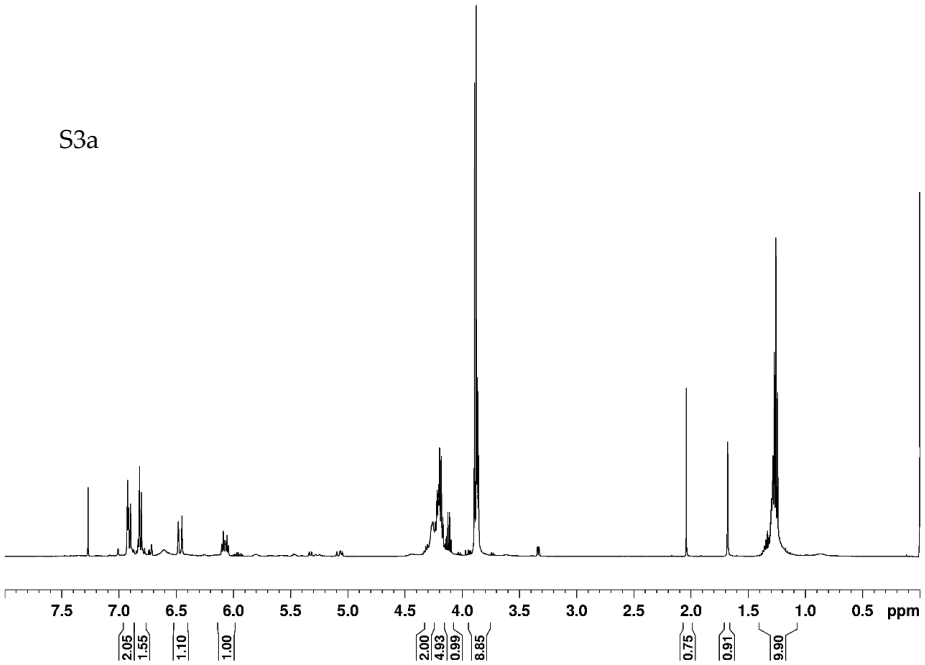
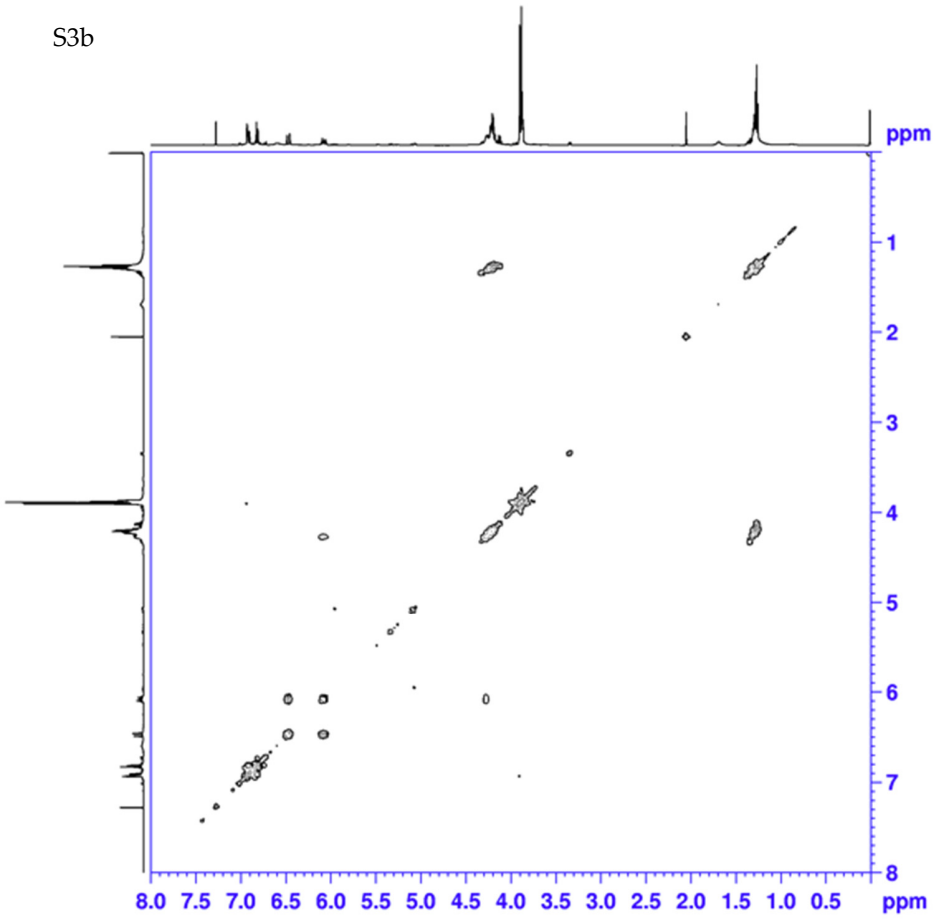


Figure S2. NMR spectra of the purified ene reaction product between allylbenzene and DEAD: a) ^{13}C spectrum (upper plot), b) ^{1}H spectrum (lower plot). The letter A denotes the ene product and the subscripts correspond to the numbering shown in Scheme 3. The letter X indicates the $CDCl_3$ peaks.

S3a


7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.05 1.55 1.10 1.00 2.00 4.93 0.99 8.85 0.75 0.91 9.90

S3b


ppm
1 2 3 4 5 6 7 8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

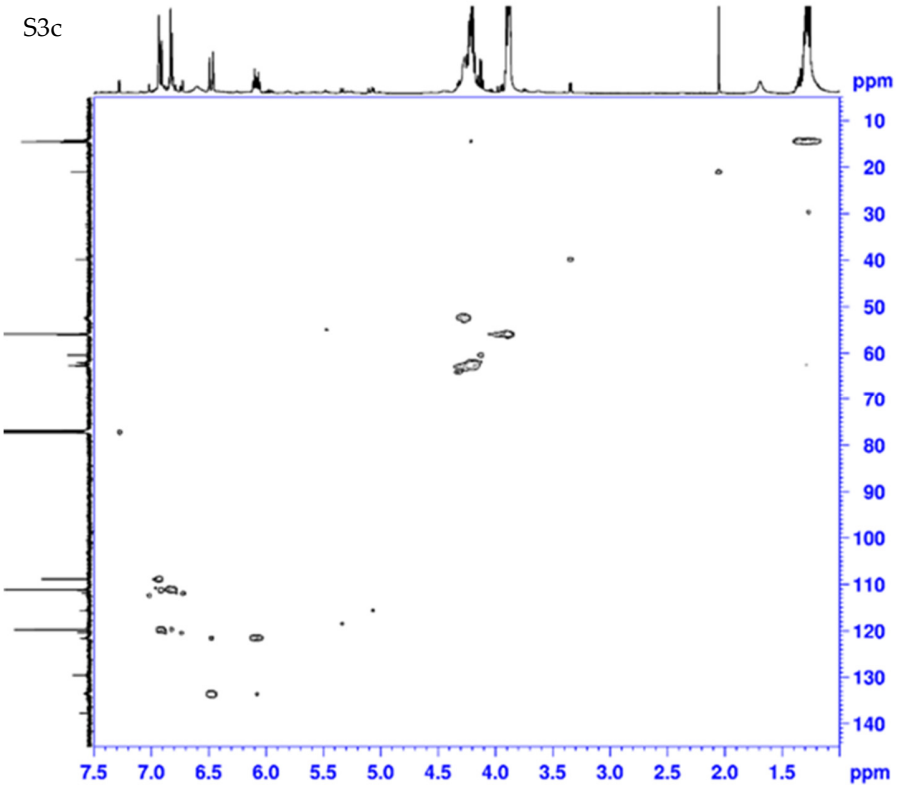


Figure S3. NMR spectra of the ene reaction products between methyl eugenol and DEAD (sample M-1): a) ^{1}H spectrum with integration; b) 2D COSY plot (^{1}H-^{1}H shift correlation); c) 2D HSQC (^{1}H-^{13}C shift correlation).

S4

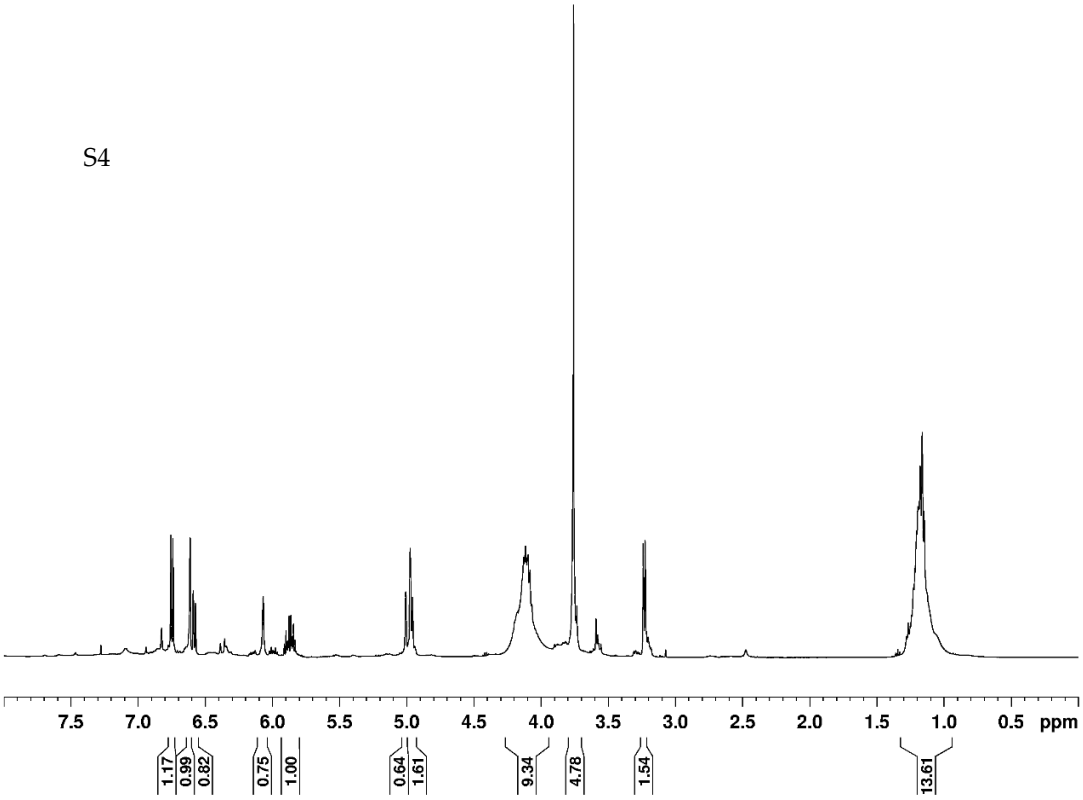


Figure S4. ^{1}H NMR spectrum (with integration) of the ene reaction products between eugenol and DEAD (sample E-1).