Orthogonal experimental design for optimizing the synthesis of vinyl-tris(2,2,2-trifluoroethoxy)silane

Jiang Pan1 Dong Hong1 Luan Wengen1 Chen Daowei2 Zheng Yunfeng2 Huang Liangbing2 Qu Zhirong1 Wu Chuan1\*

1Key Laboratory of Organosilicon Chemistry and Material Technology, Ministry of Education, Hangzhou Normal University, Hangzhou 311121. 2Novel Silicone Research Institute Affiliated to Kaihua Synthetic Material Co. Ltd., Kaihua 324300 )

E-mail: chaunwu@yahoo.com (C. Wu).

**Supplemental Materials**

The yield of Vinyl-tris(2,2,2-trifluoroethoxy)silane (VTTFEOS) was influenced by four factors with different levels. Fourier transform infrared spectroscopy (FT-IR), gas chromatography-mass spectrometry (GC-MS) and nuclear magnetic resonance (NMR) were presented. A four-factor three-level orthogonal test table was designed and experiments were conducted based on this method. Variance analysis of orthogonal experimental results was conducted and the optimal experimental conditions were verified. Other physical properties like density and the refractive indexes of VTTFEOS at 20 and 25 °C were reported.



**Figure S 1:** Yield of VTTFEOS influenced by four factors with different levels



**Figure S 2:** FT-IR spectra of VTTFEOS

![C:\Users\94395\Desktop\O1R4KU]OZMKD`@`~OTZET35.png]()

**Figure S 3:** Mass spectra of VTTFEOS



**Figure S 4:** 1H NMR spectra of VTTFEOS



**Figure S 5:** 13C NMR spectra of VTTFEOS



**Figure S 6:** 19F NMR spectra of VTTFEOS

**Table S 1:** Four-factor and three-level orthogonal tests

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  Factor Namelevel | Feeding molar ratio(A) | Oil bath temp.(B)/  °C | Feeding time of TFE(C)/ min | Condensation temp.(D) / °C |
| Level 1 | 1: 3.0 | 30 | 60 | 0 |
| Level 2 | 1: 3.25 | 50 | 120 | -10 |
| Level 3 | 1: 3.5 | 70 | 180 | -20 |

**Table S 2:** Results of orthogonal experiments

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Factor name | Feeding molar ratio | Oil bath temp/ °C | Feeding time of TFE / min | Condensation temp / °C | Yield/% |
| n.1 | 1: 3 | 30 | 60 | 0 | 89.6 |
| n.2 | 1: 3 | 50 | 120 | -10 | 91.6 |
| n.3 | 1: 3 | 70 | 180 | -20 | 92.3 |
| n.4 | 1: 3.25 | 30 | 120 | -20 | 92.3 |
| n.5 | 1: 3.25 | 50 | 180 | 0 | 90.4 |
| n.6 | 1: 3.25 | 70 | 60 | -10 | 94.5 |
| n.7 | 1: 3.5 | 30 | 180 | -10 | 98.2 |
| n.8 | 1: 3.5 | 50 | 60 | -20 | 98.6 |
| n.9 | 1: 3.5 | 70 | 120 | 0 | 92.0 |
| K1 | 91.167 | 93.367 | 94.233 | 90.667 |  |
| K2 | 92.400 | 93.533 | 91.967 | 94.767 |  |
| K3 | 96.267 | 92.933 | 93.633 | 94.400 |  |
| R | 5.100 | 0.600 | 2.200 | 4.100 |  |

**Table S 3:** Variance analysis of orthogonal experimental results (α=0.05)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Factor name | Sum of squared deviation | Degree of freedom | Mean square | F value | F0.05(2, 2) | Significance |
| Feeding molar ratio | 48.682 | 2 | 24.341 | 49.879 | 19.000 | Significant |
| Oil bath temp | 0.976 | 2 | 0.488 | 1.000 | 19.000 | Insignificant |
| Feeding time of TFE | 9.209 | 2 | 4.6045 | 9.435 | 19.000 | Insignificant |
| Condensation temp | 34.082 | 2 | 17.041 | 34.92 | 19.000 | Significant |
| Error | 0.98 | 2 |  |  |  |  |

**Table S 4:** Verification results of optimal experimental conditions

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Entry | Feeding molar ratio | Oil bath temp / ºC | Feeding time of TFE/min | Condensation temp / ºC | Yield /% |
| 1 | 1:3.5 | 50 | 60 | -10 | 98.3 |
| 2 | 1:3.5 | 50 | 60 | -10 | 98.5 |
| 3 | 1:3.5 | 50 | 60 | -10 | 97.9 |

**Table S 5:** The densities and the refractive indexes of VTTFEOS at 20 and 25 °C

|  |  |  |
| --- | --- | --- |
| Temp / °C | refraction index | density/g.cm-3 |
| 20 | 1.32427 | 1.38338 |
| 25 | 1.32212 | 1.37454 |