

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

First Named Inventor:	Willard E. Wood	
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Title:	CYCLODEXTRIN COMPOSITIONS, ARTICLES, AND METHODS	Group Art Unit: 1717

INVENTOR'S DECLARATION UNDER 37 CFR § 1.132

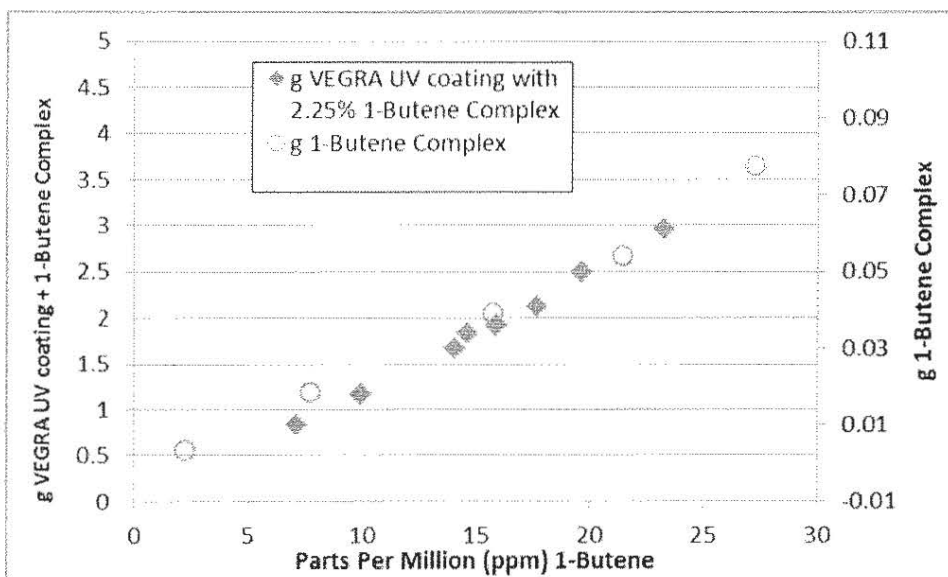
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1. I am Joseph S. Keute. I am employed at Cellresin Technologies, L.L.C., a Minnesota corporation located in White Bear Lake, Minnesota, as Senior Scientist. I have been employed at Cellresin since September 2005.
2. I graduated from St. John's University (Minnesota, 1975) with a B.S. in Chemistry and from Colorado University (Boulder, 1979) with a Ph.D. in Physical Organic Chemistry.
3. I have significant expertise in the area of cyclodextrin technology, separation technology and coating technology. I have 35 years of industrial experience in chemical products and related areas.
4. I was involved in the initial research leading to the presently claimed invention and remain involved in ongoing experimentation related to the invention. In experimental work I often use 1-butene as a surrogate for 1-MCP since both compounds are gases, and have similar properties of ethylene generating inhibitory capacity, molecular size, and other olefinic hydrocarbon characteristics; however, 1-butene does not autopolymerize explosively. Additionally, my experience has shown that both 1-butene and 1-MCP readily form CD complexes, and that the complexes have virtually identical properties of thermal and hydrolytic stability.
5. **In a set of experiments** I conducted to measure stability of the complex of 1-butene in CD (1-butene/c/CD) in radiation polymerizable monomers, I blended 1-butene/c/CD powder with UV Coating VP 10169 MF-2 High Gloss (obtained from VEGRA GmbH of Aschau am Inn, Germany; contains acrylate esters and a UV photoinitiator) to form a mixture having 2.25 wt%

1-butene/c/CD. I stored this mixture in a sealed opaque black container in the lab (about 24°C). The remaining 1-butene/c/CD powder was stored separately in a sealed container. After about 24 months of storage, I agitated the 2.25 wt% 1-butene/c/CD mixture briefly, then removed a selected weight of the mixture and combined it with 3 mL deionized water in a 250 mL glass bottle equipped with a septum cap. I placed the bottle on a lab shaker for 1 hour, then removed 1 mL of headspace gas by syringe and diluted it to a total of 250 mL with air. Then I measured the amount of 1-butene in the diluted headspace sample by gas chromatography, wherein the GC column having FID detection was previously calibrated with a 6-point 1-butene calibration curve. I tested five different weight amounts of the 2.25 wt% 1-butene/c/CD mixture in this way.

6. I repeated the above experiment with various amounts of 1-butene/c/CD powder alone, by removing selected amounts of the complex from the original container and placing each weighed sample in a bottle with 3 mL water, placing it on the lab shaker for one hour, then sampling the headspace in the same manner as for the 2.25 wt% 1-butene/c/CD mixture above.

7. I plotted the results of the two sets of headspace analyses together, as shown below. On the right ordinate, the weight of 1-butene/c/CD powder tested, which corresponds to the amount of 1-butene/c/CD in the 2.25 wt% 1-butene/c/CD mixture. On the left ordinate; that is, the ordinates were coordinated to reflect the amount of 1-butene/c/CD. The abscissa represents the amount of 1-butene measured in the headspace by GC analysis in parts per million (ppm).



8. The data demonstrate that the amount of 1-butene/c/CD present in unirradiated, 24 month stored coating is the same, within detectible limits, to the amount of the 1-butene/c/CD present in the dry powder; that is, I observed **no measurable loss of 1-butene from 1-butene/c/CD attributable to the presence of the complex in radiation polymerizable monomers.**

9. **In a second set of experiments**, I placed 1g of the 24-month stored mixture from above (2.25 wt% 1-butene/c/CD powder in VEGRA UV Coating VP 10169 MF-2 High Gloss) into a 250 mL jar and sealed the jar with a septum lid. I allowed the jar to sit in the lab for an hour; then I removed a sample of the headspace gas via syringe and measured the amount of 1-butene in the headspace by gas chromatography, wherein the GC column was calibrated in the same manner as above. I re-measured the headspace of the jar after 24 hours. Then I repeated the experiment with a second 1g aliquot of the 2.25 wt% 1-butene/c/CD mixture and analyzed the headspace after 1 and 24 hours. No 1-butene was detected in any of the four GC analyses.

10. I repeated the experiment of 9., except that after the 2.25 wt% 1-butene/c/CD mixture was placed in the jar and sealed, I irradiated the sample by placing the jar about 10 cm beneath a medium pressure mercury arc lamp operating at 79 watts per cm for about 3 seconds. The contents of the jar stopped flowing and appeared solid. After the irradiation I allowed the jar to sit in the lab for an hour, then I removed a sample of the headspace gas via syringe measured the amount of 1-butene in the headspace by gas chromatography in the same manner as in 9. I re-measured the headspace of the jar after 24 hours. Then I repeated the experiment with a second 1g aliquot of the 2.25 wt% 1-butene/c/CD mixture and analyzed the headspace after 1 and 24 hours. No 1-butene was detected in any of the four GC analyses.

11. The data demonstrate that there is **no measurable loss of 1-butene from 1-butene/c/CD attributable to either UV irradiation or the resulting polymerization reaction of radiation polymerizable monomers with which 1-butene/c/CD is mixed.**

12. I hereby declare that all statements made herein of my own knowledge and all statements made on information and belief are believed to be true; and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the present application, or any patent issued thereon.

Date: 2/14/2014

by: Joseph S. Keute
Joseph S. Keute,
Cellresin Technologies, L.L.C

Subscribed and sworn to before me
this 14th day of February, 2014

Connie Prasher

Notary Public

