Apeiron synthesis

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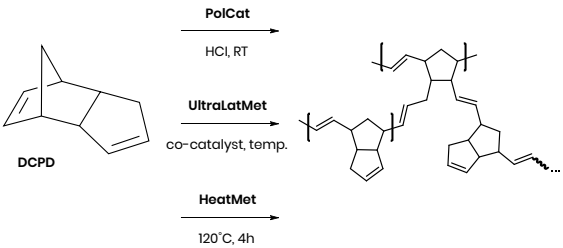
From Ideas to Polymers: Apeiron Synthesis Paving the Way



Apeiron has developed a wide portfolio of latent catalysts dedicated to Ring Opening Metathesis Polymetization (ROMP) reaction. Depending on the activation mode, the catalysts can be divided into three groups:

- thermally activated catalysts such as HeatMet,
- acid activated catalysts such as PolCat,
- catalysts activated with metal complexes such as UltraLatMet.

Leveraging our broad product portfolio and years of experience, we create sophisticated catalytic systems comprising catalysts, activators and/or retardants. Fine tuning of formulation composition allows for precise manipulation of its key properties.

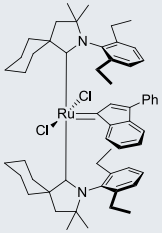
- formulations containing our catalytic systems are characterized by a long shelf life of up to 6 months and highly controllable activity,
- gelation times range from few seconds to two months at room temperature.

Compatibility

Formulations are compatible with many different fillers (glass fiber, carbon fiber, graphite, ceramics, mineral flame retardants) which enables preparation of composite materials with many desired properties: increased mechanical strength, high thermal conductivity, improved electrical and/or fire resistance. Our formulations are tailored for applications involving norbornene, dicyclopentadiene, and their derivatives.

Production

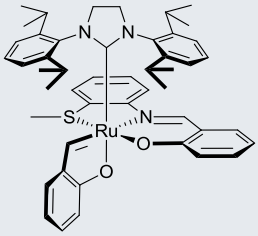
Our catalytic systems allow for the production of low odor polymers that can be used in the production of car interiors. Catalysts compatible with aqueous media enable polypeptide synthesis, DNA and mRNA modification, as well as preparation of specialty polymers.



UltraLatMet

AS2098 2501978-79-4

Patent number:
PCT/IB2016/054486, WO2006138166



PolCat

AS2151 2586056-16-6

Patent number:
PCT/IB2022/050893

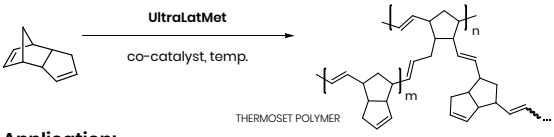
UltraLatMet -D1

AS2098-D1

soluble in monomer
(352 mg + 10 g DCPD)

shelf life:
30 days at 23˚C
6 months at 5˚C

UltraLatMet catalyst

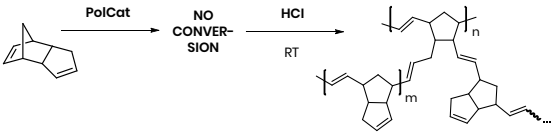


Application:

- latent catalyst for ROMP; optimal performance requires co-catalyst,
- shows excellent shelf life (up to 6 months) in dicyclopentadiene in the presence of polymerization retardant,
- allows preparation of one-component formulation with shelf life of up to 60 days,
- high stability allows catalyst handling on air, adjustable gelation time ranging from seconds to days.

R. Gawin, A. Kozakiewicz, P. A. Guńka, P. Dąbrowski, K. Skowerski "Bis (Cyclic Alkyl Amino Carbene) Ruthenium Complexes: A Versatile, Highly Efficient Tool for Olefin Metathesis" Angew. Chem. Int. Ed, 56, 910, (2017).

PolCat catalyst



Application:

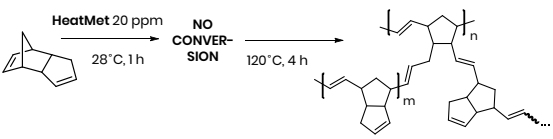
- latent catalyst for ROMP activated by hydrogen chloride,
- excellent latency and shelf life – solutions of PolCat (20-200 mol ppm) in monomers (DCPD, norbornene and their derivatives) can be stored at room temperature for 3-6 months,
- high activity after activation – short gellation time , 1-5 s,
- usually used in two formulations system – PolCat in monomer is mixed with dry HCl in monomer in the ratio of 1:1-10:1, v/v.

UltraLatMet-D1 catalyst

As a solution in toluene:
Very easy soluble in toluene but precipitate is forming after time. As a stock solution of catalyst must be used within several hours.

As a solution in dicyclopentadiene:
It takes ca. 10-20 minutes of mixing to be fully dissolved. The solution is insensitive to air and moisture but for longer storage it is recommended to keep solution under inert gas atmosphere (e.g. argon) in a cool place. After preparation the viscosity of a solution will increase slowly. It will be usable for 1 month at room temperature and 6 months at 5˚C.

HeatMet catalyst



>99%

Polymer characteristics:
R_m maximum straiin [MPa] = 46
E Young's modulus [GPa] = 1.66

Latent, thermally activated catalyst for ROMP. Activation temp. 80-90°C. High stability allows catalyst handling on air.

References: S. J. Czarnocki, I. Czelusniak, T. K. Olszewski, M. Malinska, K. Wozniak, K. Grela "Rational and Then Serendipitous Formation of Aza Analogues of Hoveyda-Type Catalysts Containing a Chelating Ester Group Leading to a Polymerization Catalyst Family" ACS Catal., 7, 4115 - 4121, (2017).
Additional References: J. A. Herman, M. E. Seazzu, L. G. Hughes, D. R. Wheeler, C. M. Washburn, B. H. Jones "Depolymerization of Cross-Linked Polybutadiene Networks in Situ Using Latent Alkene Metathesis" ACS Appl. Polym. Mater., 8, 2177-2188, (2019).

co-catalyst C6

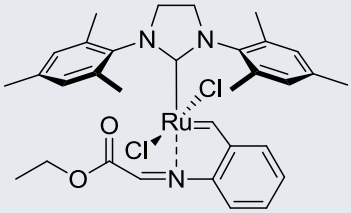
As a solution in dicyclopentadiene:
It takes a few minutes of mixing to be fully dissolved and can be heated up to speed up the dissolving. Keep under inert gas during dissolving process. The solution is a little sensitive to air and moisture during storage, but no evidence of decomposition was observed after 24h of exposure to air. No evidence of decomposition was observed after 6 months when kept in dark under argon atmosphere.

co-catalyst C7

As a solution in toluene:
Very easy soluble and stable in toluene. No evidence of decomposition was observed.
As a solution in dicyclopentadiene:
It takes a few minutes of mixing to be fully dissolved and can be heated up to speed up the dissolving. The solution is insensitive to air and moisture but for longer storage it is recommended to keep solution under inert gas atmosphere (eg. argon). No evidence of decomposition was observed after 6 months.

co-catalyst C8

As a solution in toluene:
It takes a few minutes of mixing to be fully dissolved and can be heated up to speed up the dissolving. No evidence of decomposition was observed.
As a solution in dicyclopentadiene:
It takes a few minutes of mixing to be fully dissolved and can be heated up to speed up the dissolving. The solution is insensitive to air and moisture but for longer storage it is recommended to keep the solution under inert gas atmosphere (e.g. argon). No evidence of decomposition was observed after 6 months.



HeatMet

AS2055 2248443-33-4

Patent number:
PCT/IB2013/002543

C6

soluble in monomer
(35 mg + 10 g DCPD)

shelf life:
6 months at 23°C

C7

soluble in monomer
(190 mg + 10 g DCPD)

shelf life:
6 months at 23°C

C8

soluble in monomer
(190 mg + 10 g DCPD)

shelf life:
6 months at 23°C

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AS4028

soluble in tetrahydrofurane
(325 mg + 1 ml THF)

shelf life:
6 months at 23˚C

catalyst-co-catalyst system

Table 1.
Polymerization conditions depending on the selected catalyst-co-catalyst system.

Monomer	Formulation composition for 1 kg of monomer	Mold temp.* [°C]	Gel time** [min:sec]	Curing time [min:sec]
Dicyclopentadiene (the results were obtained for Ultrene 99-6 dicyclopentadiene monomer)	180 mg AS2098-D1 + 190 mg co-cat C8	23	>60 days	-
		80	41:00	-
		90	13:00	33:00
	180 mg AS2098-D1 + 190mg co-cat C7	23	120:00	-
		80	5:30	13:00
	180 mg AS2098-D1 + 17,5 mg co-cat C6	23	1:20	2:20
		50	0:14	1:30
	550 mg AS4028	23	2:20	5:30
		50	0:40	1:50

* 10 g of formulation in glass vial in oil bath
** gel time and curing time depends on monomer purity

Properties for pure pDCPD:

- HDT (1.8 MPa load) = 40-140˚C (190 possible)
- tensile strength = up to 55 MPa
- elongation = 4-7 % at yield
- elongation at break = 4-50%
- flexural strength = up to 78 MPa
- thermal insulation = 0.17 W/mK
- dielectric strength = 33 kV/mm
- dielectric constant = 2,5 ε
- Shore hardness = up to 87 D

Properties can be modified by incorporating other monomers and/or fillers, additives:

- Mineral fillers (fumed silica, Wollastonite, Silatherm, graphite, flame retardants)
- Fibers (glass, carbon, aramid etc.)
- Antioxidants (eg. BHT, Irganox)
- Plastifiers (eg. EPDM)
- Monomers: tricyclopentadiene, cyclooctadiene, ethylidene norbornene, norbornene, Divinylbenzene.