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Product Information

RAFT Agents

Catalog Numbers **722987, 723274, 723002, 722995, 723037, 723010, 723029**
Technical Bulletin AL-264

TECHNICAL BULLETIN

Product Description

RAFT (**R**eversible **A**ddition-**F**ragmentation chain **T**ransfer) is a form of living radical polymerization involving conventional free radical polymerization of a substituted monomer in the presence of a suitable chain transfer (RAFT) reagent. Use of the appropriate RAFT agent and polymerization conditions allows for the synthesis of polymers with low PDI and high functionality. For a further explanation of RAFT technology and its advantages, please visit sigmaaldrich.com/materialmatters to see Material Matters Vol. 5, No 1 on Modern Polymerization Techniques.

Reagents

RAFT Agent	Catalog Number
2-Cyano-2-propyl benzodithioate	722987
4-Cyano-4-[(dodecylsulfanylthiocarbonyl) sulfanyl]pentanoic acid	723274
Cyanomethyl methyl(phenyl) carbamodithioate	723002
4-Cyano-4-(phenylcarbonothioylthio) pentanoic acid	722995
2-Cyano-2-propyl dodecyl trithiocarbonate	723037
2-(Dodecylthiocarbonothioylthio)-2-methylpropionic acid	723010
Cyanomethyl dodecyl trithiocarbonate	723029

Precautions and Disclaimer

These products are for R&D use only, not for drug, household, or other uses. Please consult the Material Safety Data Sheet for information regarding hazards and safe handling practices.

Storage/Stability

723274 - Store the product at –20 °C and keep tightly closed. The product is light sensitive.

722995 and 723037 - Store the products at 2–8 °C and keep tightly closed.

722987, 723002, 723010, and 723029 - Store the products at 2–8 °C and keep tightly closed. The products are light sensitive.

Procedures

Procedures of RAFT polymerization as performed by researchers at CSIRO.

A. Methyl Methacrylate Polymerization - using methyl methacrylate (MMA, Catalog Number M55909) as the monomer, AIBN (Catalog Number 441090) as the initiator, and a RAFT agent (Catalog Number 722987 or 723274).

1. Prepare stock solution of methyl methacrylate (15 mL, 0.14 mol) and AIBN (20.1 mg, 0.122 mmol) in 5 mL of benzene.

2. Aliquot 2 mL samples of stock solution into ampules containing Catalog Number 722987 (12.3 mg, 0.056 mmol) **or** Catalog Number 723274 (22.5 mg, 0.056 mmol).
   Note: Other glassware suitable for handling air sensitive reactions, such as a schlenk reaction tube (Catalog Number Z515981), may be used as an alternative to a sealed ampule.

3. De-gas contents of ampules by three repeated freeze-evacuate-thaw cycles (0.05 mm Hg) and seal under vacuum.

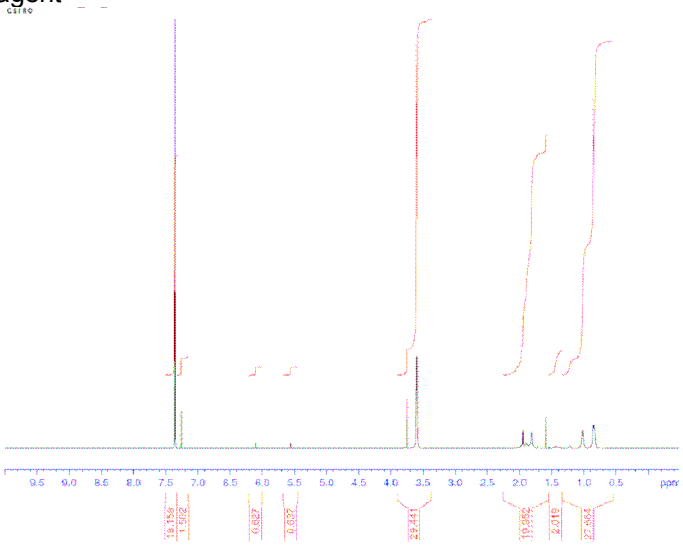
4. Polymerize by placing sealed ampule in heated oil bath (60 °C) for 15 hours.

Researchers at CSIRO have tested select Aldrich RAFT agents in the polymerization of methyl methacrylate and analyzed the resulting polymers. The data are presented in Table 1.

Table 1.
Characteristics of Poly(methyl methacrylate) synthesized by RAFT polymerization.

RAFT Agent	Polymerization Time	M_n	PDI	% Conv. (NMR)
722987	4 hours	8,468	1.14	24.5
722987	15 hours	30,032	1.07	93.6
723274	15 hours	25,959	1.08	98.5

Figure 1.
NMR of poly(methyl methacrylate) polymerized for 15 hours using Catalog Number 722987 as a RAFT agent



B. Vinyl Acetate Polymerization - using vinyl acetate (Catalog Number V1503) as the monomer, AIBN (Catalog Number 441090) as the initiator, and a RAFT agent (Catalog Number 723002).

1. Prepare solution of vinyl acetate (2.0 ml, 23.23 mmol), AIBN (2.0 mg, 0.012 mmol), and Catalog Number 723002 (26.64 mg, 0.12 mmol) in an ampule.
   Note: Other glassware suitable for handling air sensitive reactions, such as a schlenk reaction tube (Catalog Number Z515981), may be used as an alternative to a sealed ampule.

2. De-gas contents of the ampule by three repeated freeze-evacuate-thaw cycles (0.05 mm Hg) and seal under vacuum.

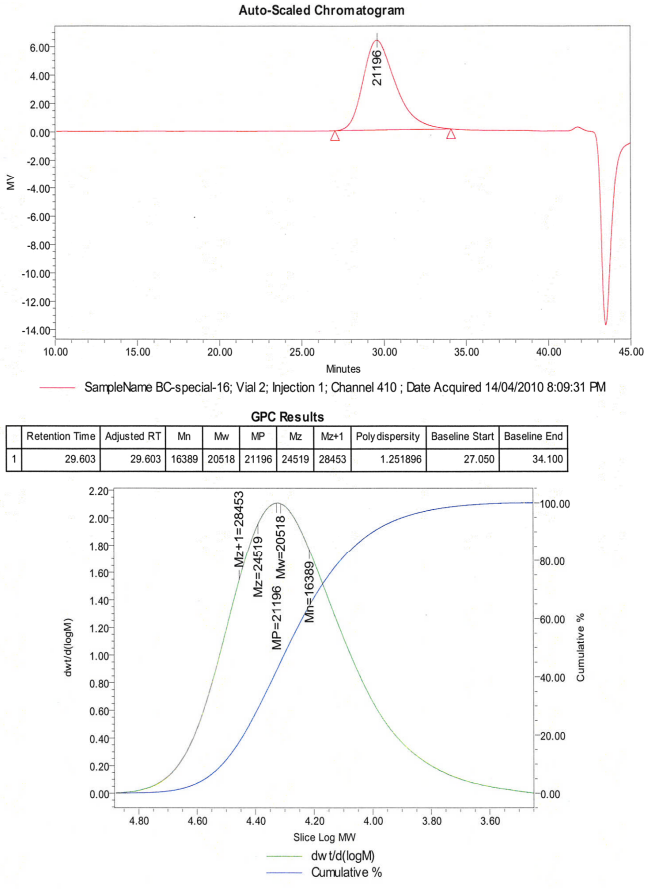
3. Polymerize by placing sealed ampule in heated oil bath (60 °C) for 16 hours.

Researchers at CSIRO have tested select Aldrich RAFT agents in the polymerization of vinyl acetate and analyzed the resulting polymer. The data are presented in Table 2.

Table 2.
Characteristics of poly(vinyl acetate) synthesized by RAFT polymerization.

RAFT Agent	Polymerization Time	M_n	PDI	% Conv. (NMR)
723002	16 hours	16,400	1.25	91

Figure 2.
GPC analysis of poly(vinyl acetate) polymerized for 16 hours using Catalog Number 723002 as a RAFT agent



	Retention Time	Adjusted RT	Mn	Mw	MP	Mz	Mz+1	Poly dispersity	Baseline Start	Baseline End
1	29.603	29.603	16389	20518	21196	24519	28453	1.251896	27.050	34.100

Patents WO98/01478 and WO99/311444
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