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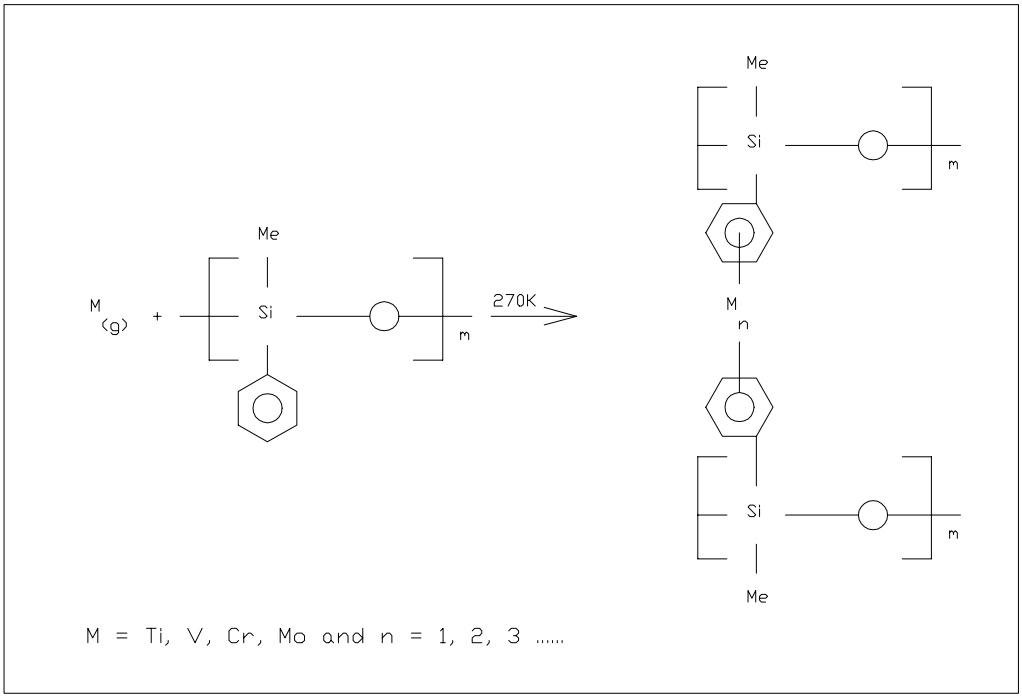
TORROVAP

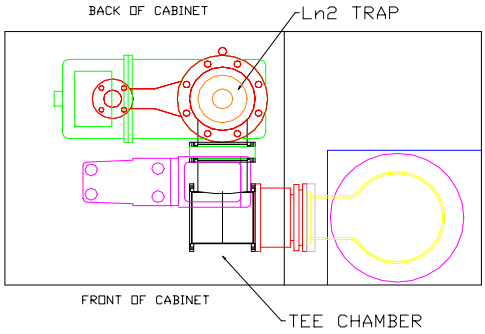
A new generation of Metal Vapour Rotary Reactor System for the Solid, Liquid or Solution Phase Synthesis of Inorganic / Organometallic Compounds and Catalysts.

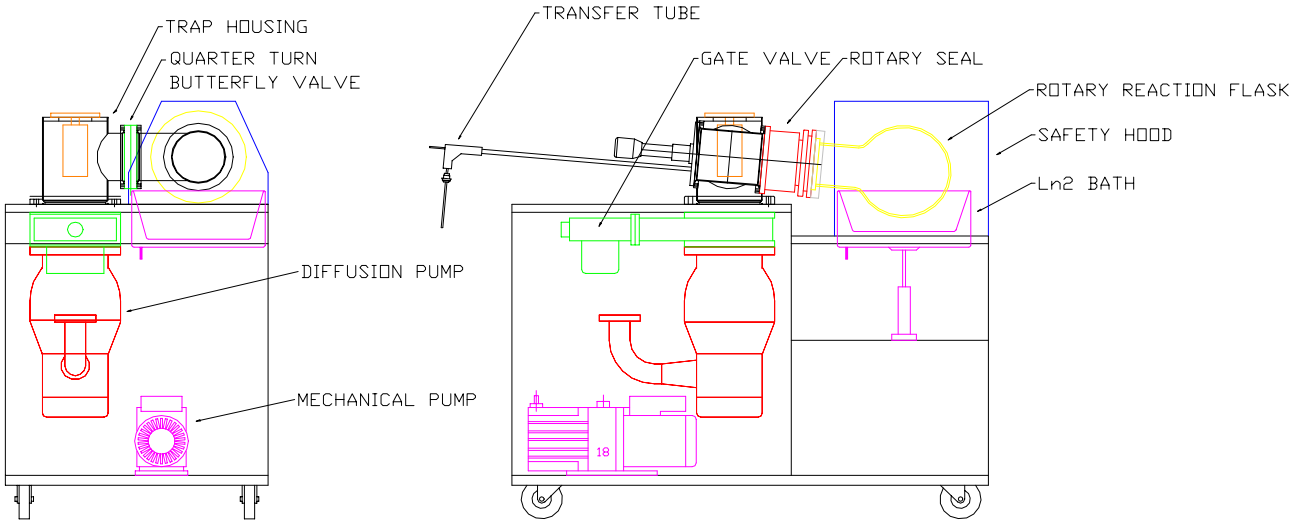


Torrovap Industries Inc. Manufacturers of Metal Vapour Synthesis, Matrix Isolation and Scientific Research Equipment.

TVP800
REACTOR CABINET
GENERAL LAYOUT
SOME COMPONENTS
HAVE BEEN
OMITTED FOR CLARITY

TOP VIEW OF EQUIPMENT





1. Diffusion pump - 2600 liters / min.
2. Demountable liquid Nitrogen cold trap
3. Quarter turn butterfly valve
4. Liquid Nitrogen cooled Transfer Tube
5. Rotary Seal
6. Temperature controlled and monitored Ligand Inlet System
7. Electrostatically focused Electron Gun (2.5 kw or 6.0 kw model)
8. Rotary Reaction Flask (5L or 12L)
9. Schlenk compatable reactant or product inlet or outlet system
10. Schlenk equipment mounting plate
11. Pneumatic driven Liquid Nitrogen Cooling Bath
12. Safety Hood with Lexan viewing area
13. Full metal cabinet with safety interlock on doors and safety hood
14. Full swivel casters for positioning

Dimensions: Approx. 64” long, 28” deep, 44” high
Services Required: Cooling water, 208/230V, 3 phase + Neutral and Ground, 30 amp electrical supply, power supply units may be altered according to end user requirements and service available. To be specified at time of order by customer.

The Torrovap 800 model Rotary Metal Vapour Synthesis equipment has now undergone a change in it's design and operating characteristics.

1.0 Design changes:
Since the original equipment was designed in the late 1980's there has been significant changes in commercially available power supplies which were not available at the time. The original power supplies which were used in the earlier models were large heavy transformers which required custom electronics to be built for the units. Since that time certain components were phased out of production and subsequently availability which led Torrovap to a re-configuration of the equipment and it's power supplies.

2.0 Operating changes:
In addition to the modernization of the power supplies we felt it was time to bring the control of the unit up to present day standards by implementing a computer control system. Torrovap has developed it's own custom software for the control of the equipment.
As in many lab's, floor space availability can be a problem and by providing computer control of the equipment we have done away with the large control cabinet which was required on past units. Another advantage which was quickly realized in the operational planning was the fact that just about anyone could operate the equipment easily with minimal training. The operational features are presented below.

2.1 Control - The unit can now be operated in two different modes.

2.1.1 - Manual mode - requires the user to direct the control of the equipment in all phases of it's operation and processing.

2.1.2 - Automatic mode - allows the user to run the equipment hands free while it performs *pre-specified processes.

2.2 Experiment Processing and Recording

2.2.1 - Auto logging or recording of manual operations is now performed within the software where the user has the ability to reproduce past experiments.

2.1.3 - * Pre-specified processes can now be entered into a "recipe" which are parameters the user pre-defines. The "recipe" can be stored for later review or repeat processing without having to reset dials and write down experimental procedures as was required in the past.

2.3 User level controls

2.3.1 - With past experience in University environments it has been found that not only can the equipment be used for teaching purposes but also for pure research. Organizations or individuals may require the need to keep their experiments and subsequent research findings a secret from other users. This can be done with user level access, certain functions for the equipment can also be locked out to by system administrators in order to prevent unauthorized operation of the equipment or operational components.

2.4 Data logging

2.4.1 - A full data logging is performed by the software which records Vacuum level, Resistive or E-Beam power settings, Quartz crystal monitoring of evaporation rates, time or process duration...

2.5 Quartz Crystal Monitoring

2.5.1 - In our previous equipment we had provided Quartz Crystal Monitoring with a single quartz crystal for either the Resistive source or the Electron gun assembly. We believe that in order to get a true and accurate measurement in material evaporation each material, in the event of dual resistive or dual electron gun operation, being evaporated should be individually monitored.
We have now incorporated a dual quartz crystal monitoring system with each crystal focused at the particular sample with virtually no possibility of cross contamination between samples being evaporated. The quartz crystal monitor can also be zeroed out for subsequent operations.

2.6 Safety Interlocks

2.6.1 - All safety interlocks have been interfaced to the monitor screen and computer control, where any failure or an interlock which has not been engaged or becomes disengaged for any reason, the user will be immediately alerted to the location of the fault.

2.6.2 - Any interlocks which have either been interrupted, disengaged or not initially connected, will prevent the equipment from becoming operational or will immediately disable operation of either the resistive mode or the high voltage mode. This feature is for the safety and protection of the user and any other personnel in the area as well as system protection.

The Torrovap apparatus is a self-contained metal vapour synthesis system designed to produce a variety of new materials such as inorganic and organometallic compounds, organic and organometal polymers and catalysts to name a few. It consists principally of a rotating reaction flask in which there is an axially mounted resistive or evaporation source. The system is maintained under high vacuum by a high speed pump assembly. Compound synthesis is achieved by two principal experimental techniques as follows:

1) Metal vapour rotary solution experiments

A quantity of liquid or solution is introduced into the rotary reaction vessel thereby creating a film of liquid over the inside wall of the flask. After the system has been evacuated, quantitative metal or salt evaporation from the vapour source commences resulting in direct reaction between atomic or molecular species of the evaporant and the liquid or solution reagent. The resulting reaction product can then be transferred anaerobically from the reaction flask for subsequent examination and experimentation.

2) Metal vapour rotary co-condensation experiments

In this technique, a temperature controlled shower head assembly mounted alongside the evaporation source is used to quantitatively spray vapours of liquid or gaseous reagents onto the liquid nitrogen cooled surface of the rotating reaction vessel while it is maintained under vacuum. Simultaneously, the high temperature source is producing a metal atom or salt vapour stream that is mixing intimately and reacting with the reagent on the wall of the vessel. The reaction products that result from this kind of co-condensation reaction are allowed to warm-up, melt down and are subsequently transferred anerobically from the reaction vessel for further investigation.

The TORROVAP system has been constructed to be entirely compatible with conventional Schlenk line, nitrogen and vacuum techniques required for handling air sensitive materials. Moreover, specially designed temperature controlled transfer tubes (300-77 K) are available on request, which are ideally suited for removing thermally labile products from the reaction vessel without decomposition.

CHEMICAL SYNTHESIS WITH METAL VAPOUR REAGENTS

SYNOPSIS. A significant development in chemical synthesis involving the control and manipulation of metal atomic and cluster chemical reagents over the temperature range 77-300 K permits access to a wide range of novel materials having unique chemical and physical properties.

The remarkable potency of metal vapours as highly versatile chemical reagents in a wide range of inorganic, organometallic and organic synthesis has been impressively demonstrated over the past decade [1]. The pioneering experiments of workers such as Timms, Skell, Klabunde, and Green in macroscale synthesis [2], and Ozin, Burdett, and Turner in matrix cryochemistry [3] have greatly contributed to the establishment and wide acceptance of metal vapour chemistry as a viable synthetic route. It has been discovered that new materials, often with unique properties, can be secured by the metal vapour route while they would be difficult, if not impossible, to obtain using conventional synthesis.

Almost twelve years of first hand expertise in cryochemistry, accrued by the University of Toronto and Bristol metal vapour groups has resulted in “Torrovap”, a highly versatile and cost effective metal vapour rotary reactor system. This state of the art metal vapour synthesis device is now being manufactured commercially by Torrovap Industries Incorporated.

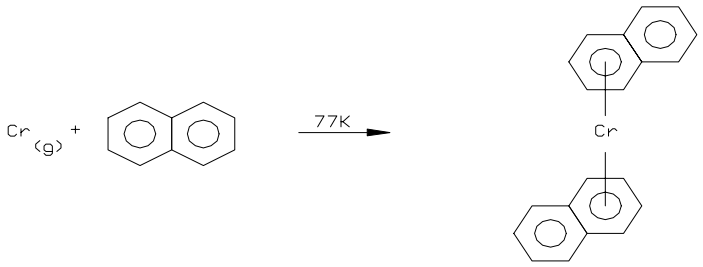
This new generation, multi-purpose rotary reactor has been designed and fabricated to allow routine use, flexibility of handling, ease of in situ metal vapour mass monitors, temperature ligand inlet system, various resistive and/or cryopumped electron gun metal vapour sources and temperature controlled products transfer tubes. Many other innovative features have all been combined and incorporated into a convenient and compact unit ideally suited for laboratory scale explanatory research production.

Some of the features that the Torrovap metal vapour reactor system offers are:

* Resistive and/or cryopumped electron gun, single or double evaporation source.
* In situ quartz crystal microbalance metal flow monitor for quantitave bimetallic application.
* Easily de-mountable rotating vacuum flask for sample material loading.
* Variable speed rotary drive mechanism.
* Range of reaction vessels (3 to 12 litres) with common vacuum sealing flanges, adapted for schlenk nitrogen and/or vacuum line handling techniques.
* Temperature controlled ligand inlet system.
* Temperature controlled product transfer tube.

The future that metal vapour or other high temperature reagents offer to the synthetic chemist, for the generation of a wide range of new materials with potentially interesting chemical, catalytic, electrical or magnetic properties, can be appreciated by a brief look at some of the chemical and technological accomplishments reported during the last few years.

(a) Condensation Experiments Using a Static Reactor



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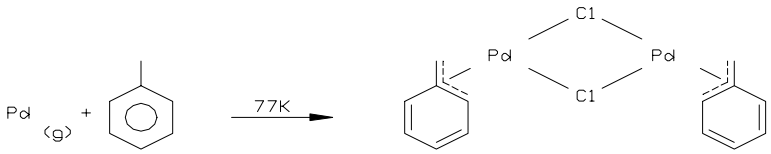

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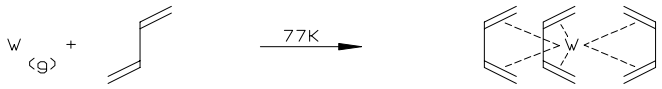

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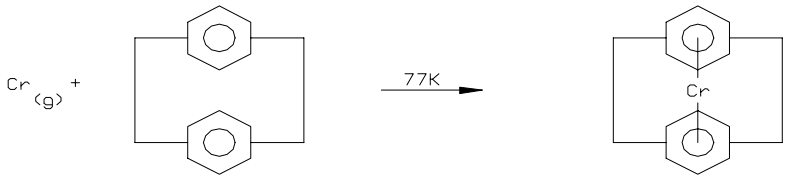

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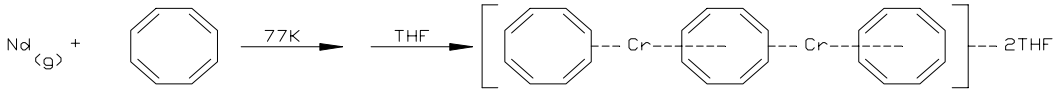

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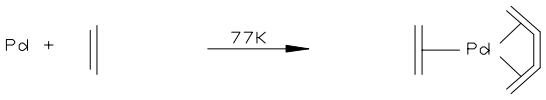

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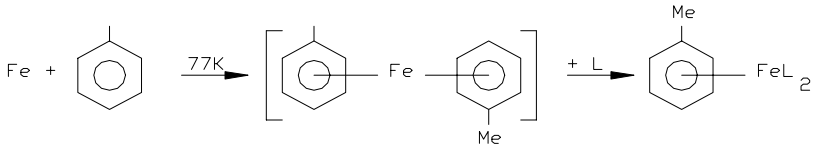
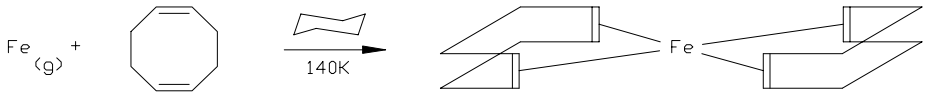


(10)

(b) Rotary Solution Reactor Experiments



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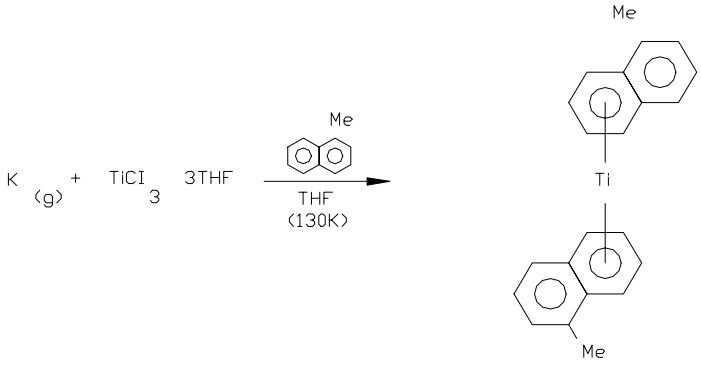

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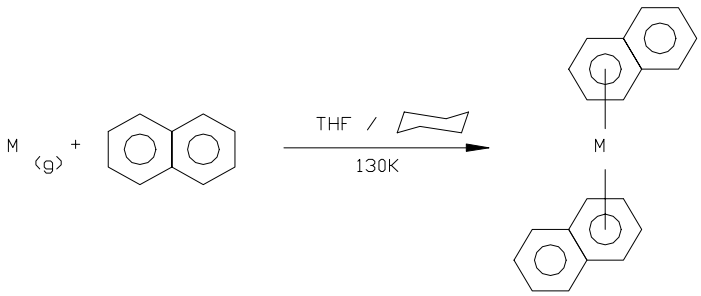
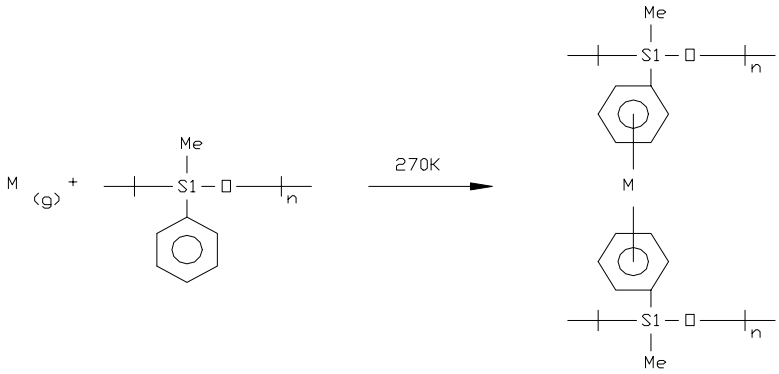


(14)

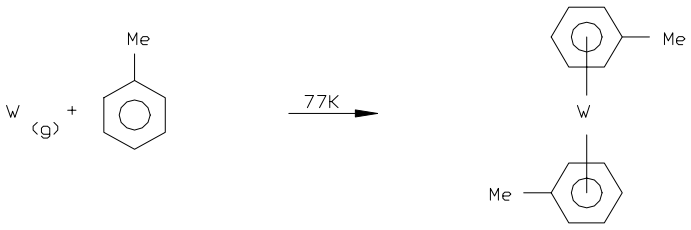
where M = Ti, V, Cr, Mo



(13)

where M = Ti, V, Cr, Mo, W

(c) Use of a Rotary Reactor in conjunction with an Electron Gun Source



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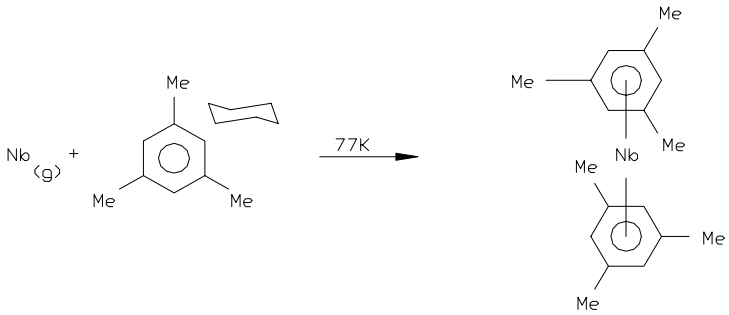
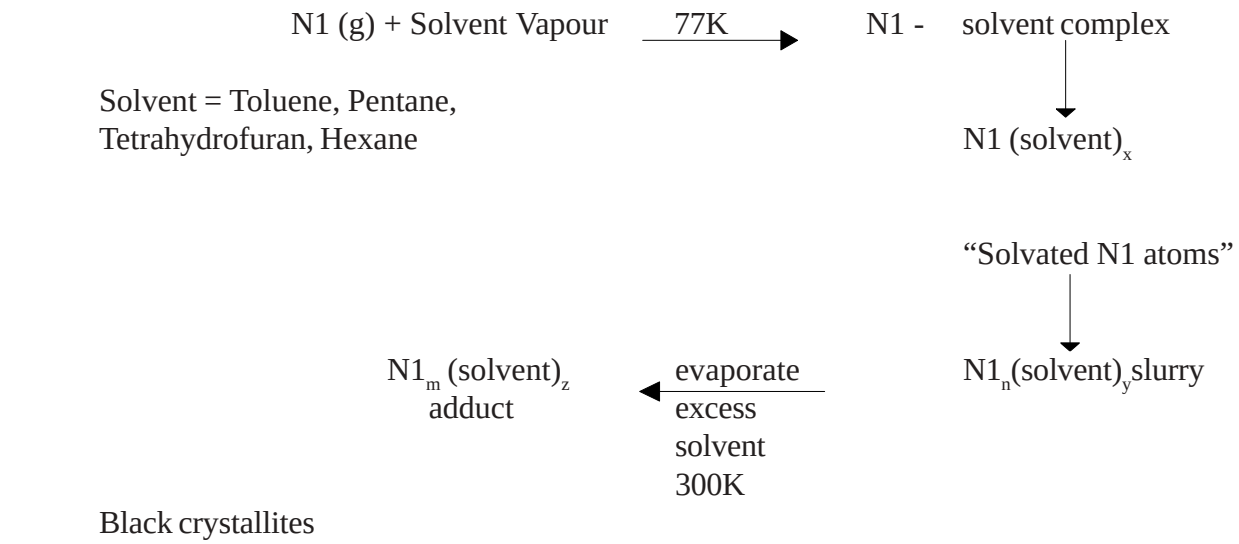
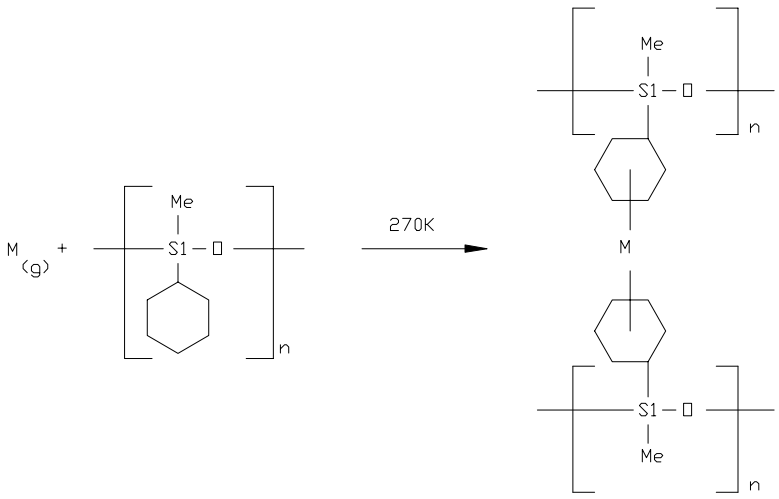


(15)

(d) Static Reactions for the Generation of Solvated Metal Atom-Metal Slurries



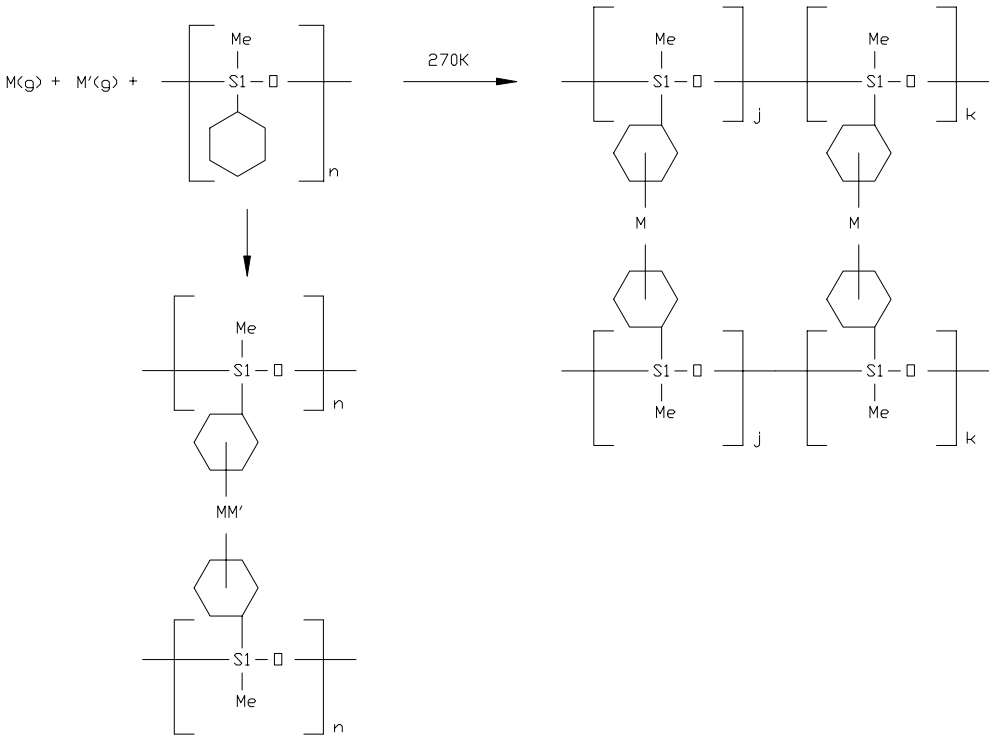
(e) Thin Static Film Metal Vapour-Liquid Polymer Experiments



(17, 18)

M = Ti, Zr, V, Nb, Ta, Cr, Mo, W, n = 1
M = Ti, V, n = 1 , 2
M = Cr; n = 1 , 2 , 3
M = Mo; n = 1 , 2 , 3 , 4 , 5

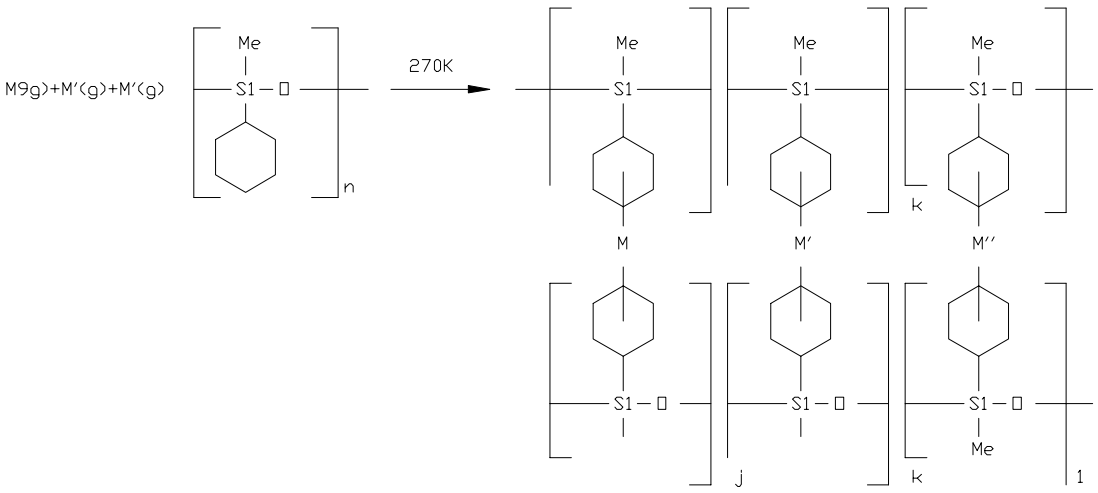
(f) Thin Static Film Bimetal Vapour - Liquid Polymenr Experiments



(19)

M, M' = Ti, Cr, or T1, Mo (19)
M, M' = V, Cr (20)

(g) Thin Static Film Trimetal Vapour Liquid Polymer Experiments



(20)

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