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TECHNICAL MEMO ON THE PURIFICATION OF TMAE

D. Anderson

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Technical Memo on the Purification of TMAE

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Particle Detector Group

When TMAE is received from the manufacturer it is much too dirty to be used. Therefore, until the material has been purified, we handle it in air when I transfer it into smaller bottles. After purification TMAE must be handled in an argon or nitrogen atmosphere since it reacts with oxygen.

There are two important steps in the purification of TMAE: 1) Washing and 2) Distillation/Drying.

Washing

The most important step in purifying TMAE is washing it with water. It is not soluble in water while its oxidation products tetramethyl oxamide and tetramethyl urea are soluble.

The water used must first have all free oxygen removed by bubbling argon or nitrogen through it. We have found the best results when this bubbling is done for a long period, such as overnight.

Under nitrogen (Note #1) the TMAE is shaken with a small portion of water and separated in a separating funnel. This is repeated until the water is clear. The TMAE is then stored in a tightly closed bottle with 4A or 5A molecular sieve to remove the water. This molecular sieve has had the oxygen removed by evacuation and backfilling with nitrogen.

For many applications the TMAE is now clean enough for use.

Distillation/Drying

Because TMAE has such a low vapor pressure (0.4 Torr at 20^{0}C and 4.9 Torr at 60^{0}C) distillation must be carried out in an oven at 50-60^{0}C. Higher temperatures may harm the TMAE.

The still used consists of two glass cells with conflat flanges, each with its own bellows value (Note #2) connected by 1/4" stainless steel tubing. A third valve is connected to a tee to allow evacuation of either or both cells. The first cell contains the TMAE. The second is filled about 1/3 full with a mixture of silica gel and molecular sieve (Note #3) which has

been baked at $400^{0}C$ under a vacuum.

The first step is to pump on the TMAE until a small fraction has been removed. IMPORTANT: a LN_2 trap must be used. The TMAE will dissolve the seals in the pump. A drawing of our LN_2 trap is included in this work.

After the first fraction is removed the second cell is placed in LN_2 and all but the last bit of TMAE is transferred. This will take several hours.

The first cell can then be replaced by a clean cell, or the bubbler to be used, and the process is reversed to transfer the TMAE. The second cell can then be baked out and is ready again to purify TMAE.

This TMAE is very clean.

NOTES

#1 This work is done in a glove box. I often use disposable glove bags (Model X-27-17) made by:

I^2R
Instruments for Research and Industry Inc.
108 Franklin Ave.
Cheltenham, PA. 19012
Phone: 215/379-3333

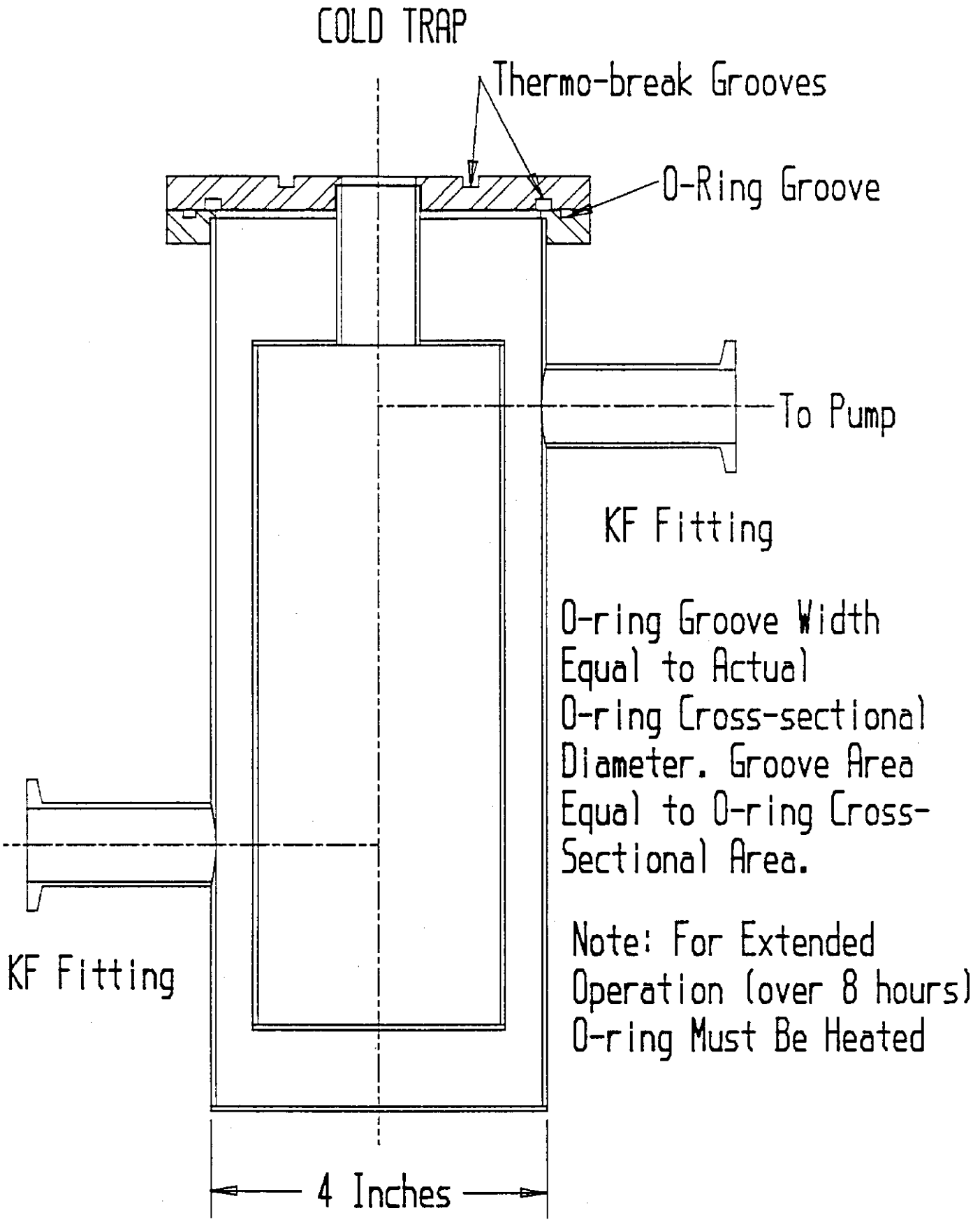
They make cleanup much easier.

#2 The glass cells we use (Model SEG-150) connect to a 2-3/4 inch flange and are made by:

MDC Vacuum Products Corp.
23842 Cabot Boulevard
Hayward, CA. 94545
Phone: 415/887-6100

The values used are "H" series bellows valves by Nupro. These are preferred because they are all metal and TMAE attacks grease and rubber.

#3 The silica gel we use is called "Chromatographic Sorbent" (S-18959-20) by Sargent-Welch.

Because the silica gel is very fine a sintered filter must be used to prevent it from being drawn into the vacuum system during cleaning. We weld a small filter

element (part SS-4F-P4-15) by Nupro on the inside of the flange. These filters also make good aerators for bubblers.