Gamma Emitting Gas Standards

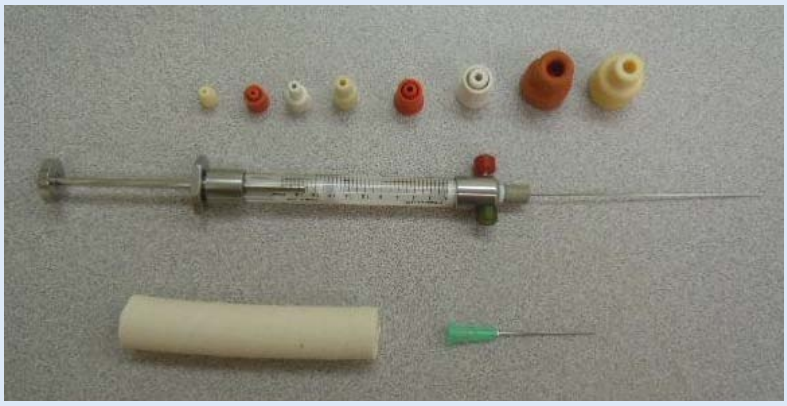
Transfer Kit Instructional Manual

Issued: 10-Jan-12

Eckert & Ziegler
Analytics

Eckert & Ziegler Analytics' Mixed Gamma Emitting Gas Standard is a mixture of Xe-133, Xe-127, and Kr-85 in air at approximately 0.85 (~86kPa) atmosphere pressure. This standard is intended for the calibration of germanium detector based gamma spectroscopy systems for gas counting in a wide variety of containers. The primary gamma ray emissions are at 81, 145, 172, 202, 375 and 514keV. Each standard is accompanied by a certificate of calibration stating the total volume of the standard spherical container, the gamma ray emission rate and the uncertainty at each energy.

Eckert & Ziegler Analytics' Mixed Gas Standard is supplied in a spherical gas counting vial with two ground glass stopcocks. This vial is designed to be counted directly, used for total transfer to large volume gas counting containers, or used for partial transfer to several smaller containers.



The transfer kit contains the necessary supplies to perform accurate volumetric transfers from the standard sphere to several gas counting geometries. Included are an assortment of rubber septa, a gas sampling syringe, and a venting needle. For gas counting containers of volume greater than one liter, total transfer of the standard is possible and can be easily performed using the small length of rubber hose included in the transfer kit and your laboratory vacuum pump.

This manual contains all instructions necessary for performing accurate volumetric transfers from the Mixed Gas Standard and example calculations for calculating the activity transferred. **Carefully read the handling precautions and gas counting considerations before proceeding!**

TOTAL TRANSFER INSTRUCTIONS

Before performing a total transfer, estimate the amount of activity needed in your container from solid or liquid efficiencies. If your calculations indicate excessive dead time, proceed with a partial transfer.

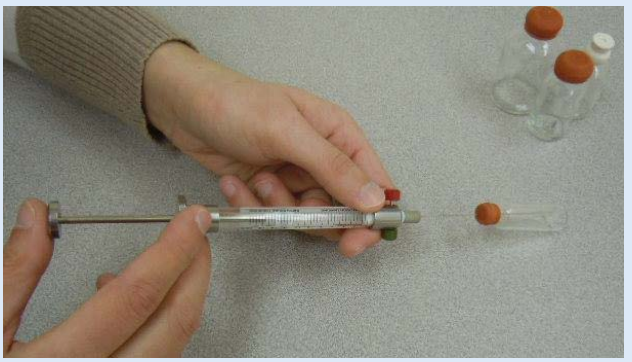
1. Check the container into which you will transfer the standard to make sure it is leak tight. Then carefully draw a partial vacuum on the container using a laboratory vacuum pump. *Be careful not to implode the container*.
2. Seal off the partially evacuated container by closing the valves or stopcocks.
3. Carefully remove the rubber retaining bands from the stopcocks on the standard sphere. *Read the handling precautions before manipulating the stopcocks.*
4. Connect the open side of the sphere to one of the valves on the counting container with the short length of hose provided.
5. Remove the rubber septum from the other side arm of the sphere.
6. Open the valve on the counting container connected to the sphere.
7. Carefully open the sphere stopcock which is connected to the counting container.

8. Fully open the other sphere stopcock and let air flush through the sphere into the counting container.
9. When the counting container has reached atmospheric pressure in several seconds, seal all valves and stopcocks and disconnect the sphere from the counting container.
10. Thoroughly mix the contents of the counting container by swirling the container.
11. Count the sphere on a gamma spectrometer to assure that all of the radioactive gas has been transferred.
12. Proceed with the gamma calibration observing the handling precautions in this manual.

PARTIAL TRANSFER INSTRUCTIONS

Before performing partial transfers, estimate the amount of activity needed in your container using solid or liquid efficiencies. You may find that total transfer to a large container results in high dead time. **If** a series of transfers are to be made from the same standard, make the transfers to small containers first and then a total transfer of the remaining gas to a large container.

1. Partial transfers must be performed using a gas tight syringe with a gas tight valve capable of retaining the standard material during transfer.



2. The gas syringe included in the transfer kit has a gas tight valve which is actuated by the red and green buttons on the syringe body. The valve is open when the green button is depressed. The valve is closed when the red button is depressed.
3. Partial transfers can be divided into two types:
   a. Transfers to containers where the syringe needle can be inserted into the body of the container during the transfer (see photo above).
   b. Transfers where the needle is prevented from reaching the body of the container (see photo below).

4. Type "b" transfers can be performed most effectively by partially evacuating the counting container, injecting a portion of the gas standard, and sweeping the standard into the body of the container with air.

PARTIAL TRANSFER INSTRUCTIONS

5. For partial transfers the standard sphere should be fitted with a rubber septum in the short side arm. Except when withdrawing gas standard material, the stopcocks should be closed and firmly held in place with the rubber retaining bands. *Read handling precautions before proceeding.*
6. To withdraw an aliquot of gas, carefully remove the rubber band from the stopcock between the septum and the sphere and open the stopcock.

7. Carefully insert the needle of the gas syringe as far as possible into the sphere.
8. Open the valve on the gas syringe by depressing the green button and pull the syringe plunger back about half way.
9. Press the plunger back in to flush the small amount of air in the needle from the syringe.

10. Pull the plunger back to withdraw exactly the desired volume of gas. Wait approximately 30 seconds to allow the pressure to equalize between the syringe and the sphere.

PARTIAL TRANSFER INSTRUCTIONS

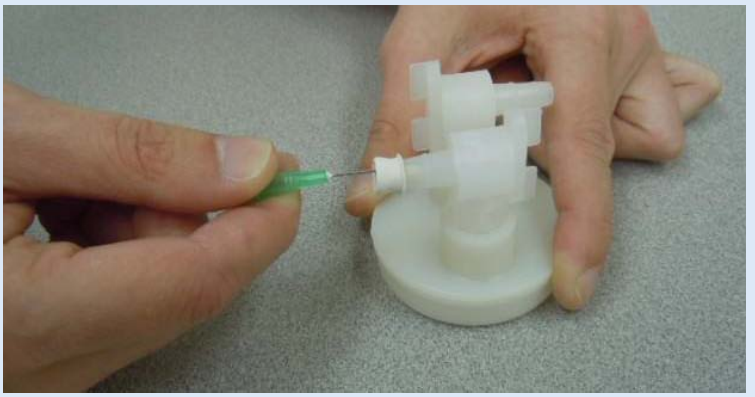
11. Close the valve by depressing the red button on the gas syringe and withdraw the needle from the septum while holding the septum in place on the standard arm. Make sure that the septum is not pulled out of the side arm when the needle is withdrawn.

12. Close the stopcock on the standard sphere and replace the rubber band.
13. The gas counting container should be fitted with a rubber septum as shown in the illustration. The counting container should be partially evacuated to avoid pressurization.
14. **If** the counting container has valves, open the valve between the septum and the body of the container so that the gas standard material may be injected.

15. Insert the gas syringe needle as far as possible into the septum on the counting container.
16. Depress the green button to open the valve on the gas syringe and push the plunger in to inject the gas standard material into the counting container. Inject all of the gas. Partial injections are not possible.
17. To completely flush the gas from the syringe into the container, withdraw the plunger about half way and push it back again while keeping the needle fully inserted in the counting container.

PARTIAL TRANSFER INSTRUCTIONS

18. Withdraw the needle from the septum, being careful not to separate the septum from the container.
19. **If** the counting container was partially evacuated before the transfer, the venting needle should be inserted in the septum and air allowed to sweep the gas standard material into the body of the container. The venting needle should not be left in the septum for more than a few seconds or back diffusion may result in loss of standard material.



20. Close all valves on the counting container.
21. Thoroughly mix the contents of the counting container by swirling the container and proceed with the gamma spectral analysis.
22. Record the volume of each aliquot of material withdrawn from the standard and record the container to which it was transferred. When performing multiple transfers the sequence of transfers should be carefully recorded since equal volumes of gas material withdrawn at different times will not contain equal activities of standard material. See sample calculations.

HANDLING PRECAUTIONS

1. Use extreme caution when manipulating the glass stopcocks on the sphere. The stopcocks are not vacuum stopcocks, and the stem can become disconnected from the body of the sphere. The stopcock stems are held in place by rubber bands for shipping and should be secured at all times except during transfers. While manipulating the stopcocks, pressure should be applied with your hands to keep the stem as far in the body as possible. As long as these simple precautions are followed, no loss of the standard should occur during stopcock manipulations.
2. When using the transfer syringe to remove an aliquot of gas from the standard, one hand should always be used to hold the rubber septum securely in the septum side arm. When withdrawing a needle from the rubber septum, there is a chance that the septum will come out of the glass side arm if the septum is not being held securely.
3. All transfers should be performed in a fume hood. All valves and stopcocks on the standard and on the counting containers should be closed before transferring containers from the hood area. All containers should be checked for leaks prior to adding radioactive gas. If the counting room or detector shield atmosphere becomes contaminated with radioactive gas, these areas should be vented to remove the gases before counting any standards.
4. Radioactive noble gases diffuse through plastic containers and rubber septa over a period of a few days. Standards prepared in plastic containers or in containers with rubber septa should be counted as soon as possible, and should be transferred to a fume hood or ventilated storage area to prevent build up of radioactive gas in the laboratory or counting room atmosphere.
5. Only gas tight syringes with a valve should be used to transfer gas standard material. Since the gas is being transferred to other than atmospheric pressure, a valve is necessary. Normal liquid handling syringes cannot be used to transfer accurately or to store gases. Regardless of the material used to construct the syringe, medical syringes are not tight enough to hold gases. A suitable tight syringe with a valve is supplied with the transfer kit.
6. **If** you reuse your gas bulb, remember that the stopcocks are individually ground and the components are not interchangeable.

GAS COUNTING CONSIDERATIONS

Efficiency curves derived from counting gas standards provide the most accurate calibration for gas counting. Unless the efficiency ratios for solid or liquid standards versus gas standards are determined at each energy and for each geometry and counting position, calculations based on solid or liquid standards can be in error by a considerable amount. Gamma ray scattering in liquids and solids causes the apparent attenuation coefficient to drop at low energies and at positions close to the detector. Because of this scattering, efficiency ratios between solid or liquid standards and gas standards will vary depending on the position of the standard relative to the detector. Calibration should be performed with gas standards in all counting positions close to the detector.

At the low energy end of the spectrum, extrapolation from the 88 keV gamma from Cd-109 in liquid or solid gamma standards down to 81 keV can be in error by as much as 10% from the extrapolation itself not even considering differences in attenuation between solids and gases. When using Ba-133 in solid or liquid sources summing errors can be significant.

The diffusion of noble gases through plastic and rubber is of great concern in gas counting. Plastic containers show considerable loss in a few days. Therefore, standards prepared in plastic containers should be counted as soon as possible after preparation. Rubber septa show a tendency to lose noble gases, especially after they have been punctured. **It** is therefore recommended that containers using rubber septa should be counted soon after preparation. Rubber gaskets and Apiezon grease have shown tendencies to absorb radioactive noble gases. Storage of radioactive noble gases in contact with rubber gaskets or hydrocarbon greases should be avoided. Silicone grease shows much less tendency to absorb radioactive noble gases.

Remember that your calculations are based on volumetric transfers which assume that the temperatures inside the bulb and the syringe are the same. Holding the syringe or the glass bulb in your hand for any length of time will change the temperature equilibrium.

CALCULATIONS FOR PARTIAL TRANSFER

When performing multiple transfers the sequence of transfers and the amount of each aliquot must be recorded even if an aliquot is not used. After each transfer the amount of material transferred should be calculated and the amount remaining in the sphere should be also calculated.

For the first partial transfer the activity transferred is given by equation (1):

$$A_T = \frac{V_A}{V_A + V_S} A_S \qquad (1)$$

Where:

A_T = activity transferred
A_S = activity in the sphere before this transfer
V_A = volume of aliquot removed from the standard
V_S = volume of standard sphere

This calculation should be performed for each isotope in the standard.

The activity remaining in the sphere is given by equation (2):

$$A_R = A_S - A_T \qquad (2)$$

If a second transfer is performed the activity transferred in the second aliquot *(*A_{T2}*)* can be calculated using equation (1) if A_S is replaced by A_R, equation (3):

$$A_{T2} = \frac{V_A}{V_A + V_S} A_R \qquad (3)$$

The activity remaining in the sphere after the second transfer will be given by equation (4):

$$A_{R2} = A_R - A_{T2} \qquad (4)$$

Additional transfer can be calculated in a similar fashion. It is very important to remember to calculate the activity remaining in the sphere after each aliquot is removed even if the aliquot is not used for counting.

LIMITED WARRANTY

Eckert & Ziegler Analytics warrants that at the time of shipment the products sold by it are free from defects in material and workmanship and conform to specifications, which accompany the product. Eckert & Ziegler Analytics makes no other warranty, expressed or implied, with respect to the products, including any warranty of merchantability or fitness for any particular purpose. Complaints of breach of warranty on radioactive products must be received in writing by Eckert & Ziegler Analytics within two half-lives of the radioactive material or 30 days, whichever first occurs. The maximum liability for any breach of warranty shall be replacement of the product or refund of the invoice price of the product. Eckert & Ziegler Analytics shall in no case be liable for special, incidental or consequential damages of any kind.