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Gas analysis — Preparation of calibration gas mixtures — Weighing methods

ADDENDUM 1

Addendum 1 to International Standard ISO 6142-1981 was developed by Technical Committee ISO/TC 158, *Analysis of gases*, and was circulated to the member bodies in March 1982.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	South Africa, Rep. of
Belgium	Italy	Thailand
Czechoslovakia	Mexico	United Kingdom
Egypt, Arab Rep. of	Netherlands	USSR
France	Poland	

No member body expressed disapproval of the document.

Annex

Precautions to be taken when weighing, handling and filling cylinders

(This annex does not form part of the standard.)

A.0 Introduction

In order to obtain the specified accuracy for this method (better than 1 % for the concentration of each component), the following precautions should be observed when weighing, handling and filling cylinders.

A.1 Accuracy of the balance

A.1.1 The precision of the balance should be consistent with the desired final accuracy.

It is assumed that the balance is of the constant-load type, and has a ratio of capacity to resolution between 10^5 and 10^7.

Typical examples of balance characteristics are given in the table.

Table — Typical examples of balance characteristics

Capacity	Resolution	Ratio of capacity to resolution
240 g	0,1 mg	$2{,}4 \times 10^6$
1 kg	0,2 mg	5×10^6
8 kg	3 mg	$2{,}7 \times 10^6$
16 kg	0,1 g	$1{,}6 \times 10^5$

Larger balances, capable of weighing full-size cylinders, may be used but should have a ratio of capacity to resolution within the desired range.

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A.1.2 Check the balance using the manufacturer's procedure if available and appropriate. Alternatively, the following procedure, which assumes that the balance uses fixed weights, the smallest of which corresponds to the range of an optical scale, may be used.

A.1.2.1 Zero the balance. Place an empty cylinder, which should be at the temperature of the balance and environment, on the balance, record its mass, then remove the cylinder. Repeat this procedure three or four times with the same cylinder.

Over a period of time comparable to that required to make a mixture by this method, the recorded masses should not vary by more than the resolution of the balance.

A.1.2.2 Place the empty cylinder on the balance. Use the zero or tare control to return the optical scale to zero. Remove the smallest fixed weight from the balance and reweigh.

If the optical scale reading is not at its maximum, within the resolution of the balance, adjust the balance sensitivity and repeat the procedure until the sensitivity is satisfactory.

A.1.2.3 Zero the balance. Place the empty cylinder on the balance, and record its mass. Add weights of class E_2 [OIML Recommendation No. 20 (1973)], corresponding approximately to the increases in mass expected when components are added to the cylinder. Record the increased masses.

The recorded increases should agree with the class E_2 weights to within the resolution of the balance.

Remove the extra weights and record the mass of the cylinder.

This should agree with the mass recorded in A.1.2.1 to within the resolution of the balance.

A.2 General handling of the cylinder

A.2.1 General

Any change in the measured mass of the cylinder, other than changes due to the addition of gases, will adversely affect the result. Potential sources of such errors are described below.

A.2.2 Changes in barometric pressure and atmospheric humidity

Changes in buoyancy caused by variations in barometric pressure should be eliminated either by weighing under vacuum or by making compensation by weighing against a reference cylinder, or by calculation (see ISO 6142, subclause 2.2.2). The amount of adsorbed water, resulting from atmospheric humidity, should reach a constant level or be subject to the same changes as affect the reference cylinder, if the test cylinder is allowed to come to thermal equilibrium after filling. For small cylinders (up to 1 kg), 1 h is sufficient. For large cylinders, the establishment of thermal equilibrium should be tested by constancy of mass.

A.2.3 Leakage from the cylinders

After the precision of weighing has been established on an empty cylinder, fill each of the cylinders to be used (with the exception of the reference cylinder, if required) to the maximum expected pressure. Allow the cylinders to come to thermal equilibrium and perform a series of weighings on each, for example during the course of a working day. Any steady loss of mass indicates a leak.

It is possible that occasional leaks may occur, due to particles of dirt becoming trapped in the valve or to other causes on addition of components. In order to detect this leakage, weigh each cylinder several times after the addition of each component.

A.2.4 Scoring or scraping of traces of metal from the cylinder, and collection of dirt or grease from the fingers on the cylinder

The effect of scratches, pick-up of dust, grease from the fingers, etc. becomes greater as the ratio of surface area to capacity of the cylinder becomes greater, i.e. it affects small cylinders more than large ones. The following handling precautions are recommended when using small cylinders. Experience will show whether they are all necessary with large ones.

Any scratches or gouges in the cylinder should be removed by means of abrasive cloth. The surface of the cylinder should be clean and polished.

During all operations, other than weighing, the cylinder should be kept in a protective sleeve. This protects the cylinder from dust, dirt, fingers, etc.

When the cylinder is transferred to or from the balance, it should be handled only by an operator wearing protective gloves.

A.2.5 Loss of traces of metal or rubber from tightening and slackening of compression fittings which connect the cylinder to the filling manifold

Connection of the cylinder to the filling manifold should, where possible, be through a compression fitting made with an "O-ring" seal. The compression nut should be slackened, not removed, to allow the filling pipe to be slid into or out of the fitting. The end of the filling pipe should be rounded and deburred so that sharp edges cannot shave pieces of rubber from the seal. The jaws of the spanner used to tighten and slacken the nut should be lined with polyethylene.

A.3 Filling of the cylinder

Carry out the filling of the cylinder as follows.

Add the components in the order of their increasing partial pressure in the final mixture. If the mixture is to contain a condensable component it may be necessary to add it at an earlier stage than that indicated by its partial pressure.

Connect the cylinder to the filling manifold and evacuate it to a pressure not greater than 10 Pa. Then, preferably, fill the cylinder to approximately $1{,}5 \times 10^5$ Pa with the complementary gas and evacuate it again to 10 Pa. Introduce the first com-

ponent to a pressure calculated according to the desired concentration (if this pressure is accidentally exceeded, remove gas from the system until the required pressure is attained). Remove the cylinder from the manifold, allow it to reach thermal equilibrium and weigh.

Thoroughly eliminate residues of the first component from the manifold and reconnect the cylinder. Introduce the second component to the desired pressure. (At no point during this operation shall the pressure in the manifold be allowed to fall below the pressure in the cylinder. As the desired pressure is approached, the total pressure should be allowed to rise slowly, and the cylinder should be isolated at the exact value required. This reduces any danger of back-diffusion of components from the cylinder.)

If further components are to be added, follow the procedure used for the second component.

Limit the concentration of any condensable component so that its partial pressure does not exceed 0,7 of its saturated vapour pressure at the lowest temperature of use.

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