INTERNATIONAL STANDARD

ISO 9894

First edition
1996-06-15

Subsampling of uranium hexafluoride in the liquid phase

Sous-échantillonnage de l'hexafluorure d'uranium en phase liquide

Reference number
ISO 9894:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9894 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Subsampling of uranium hexafluoride in the liquid phase

1 Scope

This International Standard specifies a method of subsampling suitable for taking aliquots from a representative sample of uranium hexafluoride (UF_6) in the liquid phase, the latter having come from a (UF_6) shipping container.

The subsamples are intended for isotopic analysis (1 g to 3 g), impurity analysis (10 g to 200 g) and uranium assay (5 g to 10 g).

Carbon halides, hydrocarbons and certain metal halides can be measured directly from the sample or the subsample (10 g to 30 g).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7195:1993, *Packaging of uranium hexafluoride (UF_6) for transport.*

3 Principle

After liquifying the UF_6 contained in the sample cylinder, the amount in the liquid phase which is removed by means of a buffer volume is:

— either released into a container (0,005 litre to 0,100 litre) for isotopic analysis,

— or run into a container (0 litre to 0,5 litre) for impurity analysis after hydrolysing the UF_6,

— or run into a container (0,002 litre to 0,005 litre) for uranium assay determination of UF_6.

Every precaution shall be taken to minimize the risk of isotopic and chemical contamination among the samples.

4 Reagents and liquid refrigerants

4.1 Liquid nitrogen.

4.2 Iced water at 0 °C.

4.3 Chlorine trifluoride or **nitrogen doped to 25 % with fluorine,** optional.

5 Apparatus

5.1 Metal containers

Metal containers of types 1s and 2s according to ISO 7195 and ANSI N14.1[1], or type CEA 23D are used.

5.2 Polytrifluorochloroethylene (PTFCE) containers, example given for clarification in figure 1.

5.3 Tubes in PTFCE, examples given for clarification in figures 2 and 3.

5.4 Valves (see figure 4).

1) ANSI N14.1 — 1971, *Packaging of Uranium Hexafluoride for Transport.*

5.5 Heated vacuum manifold (see figure 4)

This assembly in figure 4 does not preclude the use of other equipment which can be shown to provide an equivalent performance.

The system is installed in a heated, compartmentalized enclosure and connected to a pump unit.

The conditioning temperature is related to the function of each compartment:

90 °C to 100 °C for the liquification of the sample container to be subsampled;

80 °C to 90 °C for subsampling in the liquid phase;

65 °C to 75 °C for the aliquoting done in the gaseous phase and recovery.

5.6 Pump unit (see figure 4)

The pump unit comprises

— a liquid-nitrogen cold trap 30,

— a chemical trap 31 (NaF and Al_2O_3 of granular structure),

— a vacuum pump (rotary pump or turbo-molecular pump) 32,

— a pressure gauge.

The pumping system shall be able to pump down to 0,1 Pa in a few minutes.

For this heated vacuum manifold, the pipe lengths (internal diameter 10 mm) have been constructed as short as possible.

5.7 Dewar-type flasks, to hold refrigerants.

NOTE 1 The materials used are those most chemically inert of UF_6.

6 Operating procedure (see figure 5)

This operating procedure is applicable to the installation described in clause 5 and it guarantees that representative samples are taken.

The valves shall be operated in the order mentioned in the text.

6.1 Preparation

6.1.1 Connection of the containers

The cylinders for sampling UF_6, shall be inverted and connected to the top of a heated vacuum manifold (the collection capacity is eight sampling cylinders, 15 to 22, for this installation); connect the containers (24 or 25 and 26).

6.1.2 Pumping down

Open valves 43, 42, 41, 40, 28, 1 to 14, 23, 34 and 35 to evacuate the system, at the same time isolating the recovery container 29 by closing valves 38 and 39.

6.1.3 Pressure test

Close valve 1 to test for a pressure rise, for 3 min. The pressure shall not exceed 0,1 Pa. Close valves 5 to 14, 23, 4, 3, 34, 35 and 2.

Adjust the manifold to "heat".

6.1.4 Second pressure test

On attaining the prescribed temperatures, carry out a second test for rising pressure as follows:

Pump down (see 6.1.2). Carry out the pressure test (see 6.1.3). The pressure shall not exceed 0,1 Pa. Close valve 28. Open valves 38 and 39.

6.1.5 Passivation (optional)

The preparatory phase may be completed by passivating the system with the ClF_3 or a mixture of N_2 and F_2 (discharge from container 27).

The passivation is essential after an overhauling of the valves, pipes or pressure gauges.

6.2 Purging the lines with UF_6

NOTE 2 With turbo molecular pump no purging is needed.

The procedure is applied to each sample.

For the sample marked 15, proceed as follows.

Open the valve of sample cylinder 15, then close it. Run into the pipework by opening valves 2, 3, 4, 5, 6, 34 and 35. Wait 2 min. Trap the UF_6 in the recovery container 29 by opening valve 1 or use a spare subsample container to collect the bulk of the "purge" UF_6. Pump out the line (pressure about 0,1 Pa). Close valves 6, 5, 4, 3, 2, 34, 35 and 1.

6.3 Aliquoting into the PTFCE container for impurity analysis (marked 24)

For the sample marked 15, proceed as follows.

Open valve 5. Cool the base of container 24 with liquid nitrogen for 3 min to 4 min. Open the valve of sample container 15 then close it to isolate the amount of UF_6 to be transferred. Open valve 6 then valve 23 to carry out the run off into container 24. Close valves 6

and 23. (The operation can be repeated several times depending on the quantity of subsample required.) Check the residual pressure (≤ 1 Pa) in the line after trapping by opening valve 4. Pump by opening valves 1, 2, 3 and 23 (pressure ≤ 0,1 Pa). Close valves 23, 4, 3, 2 and 1. Disconnect container 24 which has been kept at trap temperature and set the stopper in place.

The hydrolysis operation will immediately follow the aliquoting for reasons of safety and in order to obtain representative samples. The PTFCE container can be submerged or have water poured slowly into it.

The hydrolysis operation consists of

— opening container 24 maintained at the temperature of the liquid nitrogen trap,

— hydrolysing the UF_6 by double-distilled water (5 g of H_2O per 1 g of U),

— pouring and stiring slowly to avoid spattering,

— closing the container when the reaction has finished (seeing by translucency).

6.4 Aliquoting into a 0,005 litre container for uranium assay determination (marked 25)

For the sample marked 15, proceed as follows.

Pump down. Open valve 5. Open the valve of sample cylinder 15, then close it. Open valve 6 to transfer the UF_6 up to micrometer valve 23. Close valve 6. Open the micrometer valve 23 so as to control the run-off liquid UF_6 (seeing by translucency). Close valve 23 as soon as the level of liquid in the container 25 is reached. Progressively immerse the tube in liquid nitrogen until mid-height. Open valves 4, 3 and 2 to release any liquid UF_6 remaining in the ramp (if required, take aliquots into the container marked 26, according to the procedure described in 6.5). Open valve 1 and trap the UF_6 in the recovery container 29. Evacuate the line (pressure ≤ 0,1 Pa). Close valves 4, 3, 2 and 1. Disconnect container 25 which is kept cold, and quickly plug it.

6.5 Aliquoting into the container for isotopic analysis (marked 26)

In general, this operation comes after the liquid run-off into the container 25 (marked 25) according to the procedure described in 6.4 and before releasing UF_6 into the recovery container 29.

Close valve 2. Open valves 1 and 35 (pressure < 0,1 Pa). Cool the container 26 with iced water for 5 min. Crack open valve 34 of the container 26 until the formation of UF_6 crystals is observed in the elbow (seeing by translucency). Close valves 34 and 35 after having pumped for 1 min. Open valve 2 (pressure < 0,1 Pa). Close valves 4, 3, 2, 1, 38 and 39. Open valve 28. Remove the container 26.

NOTES

3 An official form accompanies each subsample on which are recorded the origin, the quantity aliquoted, the date of aliquoting and the analyses to be carried out.

4 The whole system (manifold, containers) should be kept absolutely clean.

5 The metal or PTFCE containers are cleaned out after each use. The inner surface finishing of the containers is polished.

6 The containers used for the aliquots designated for isotopic analysis should be used for this purpose only. They are passivated after each use.

7 The hydrolysis operation is carried out under a hood, in a very clean and properly ventilated area supplied with an absolute filter.

Dimensions in millimetres

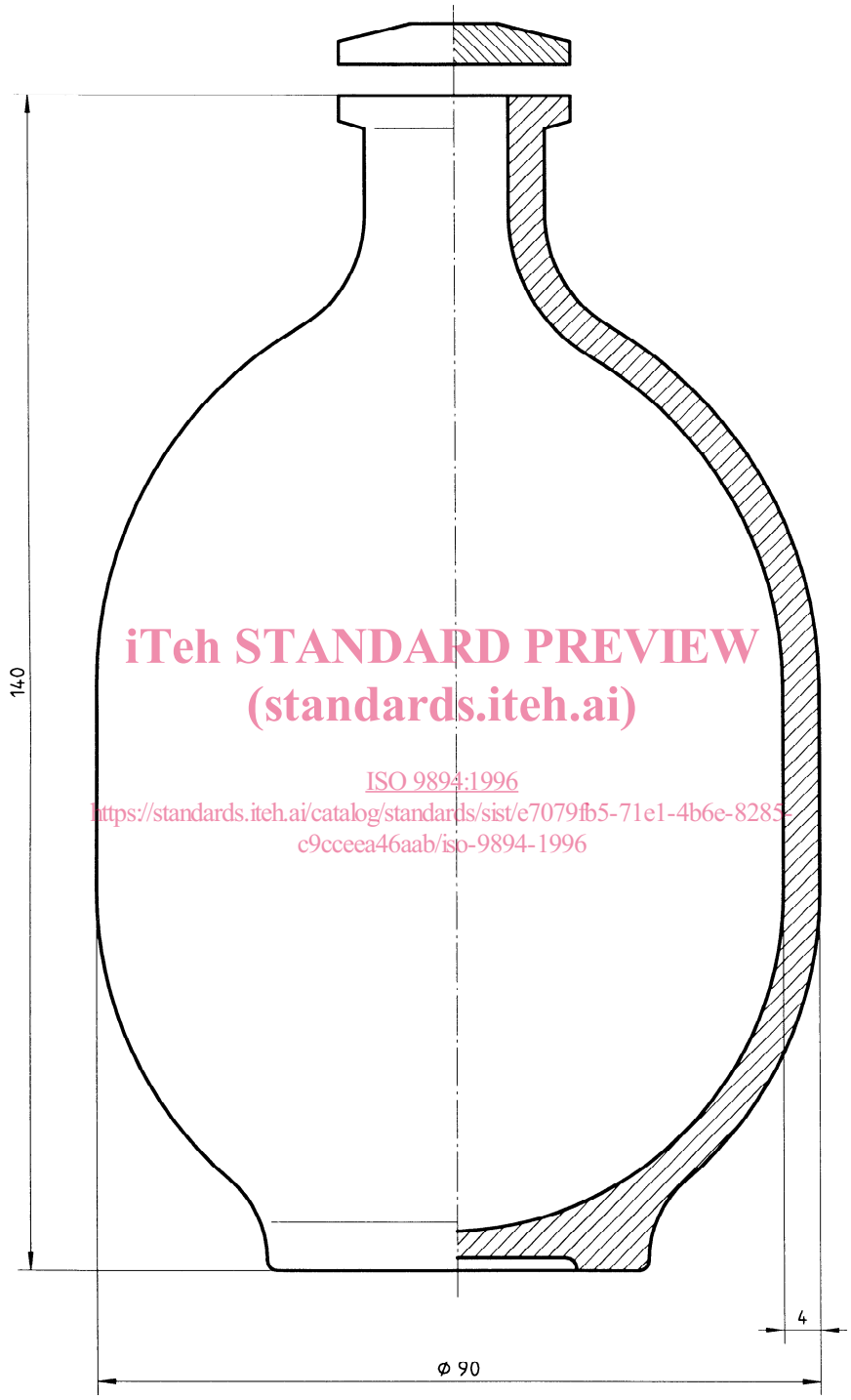
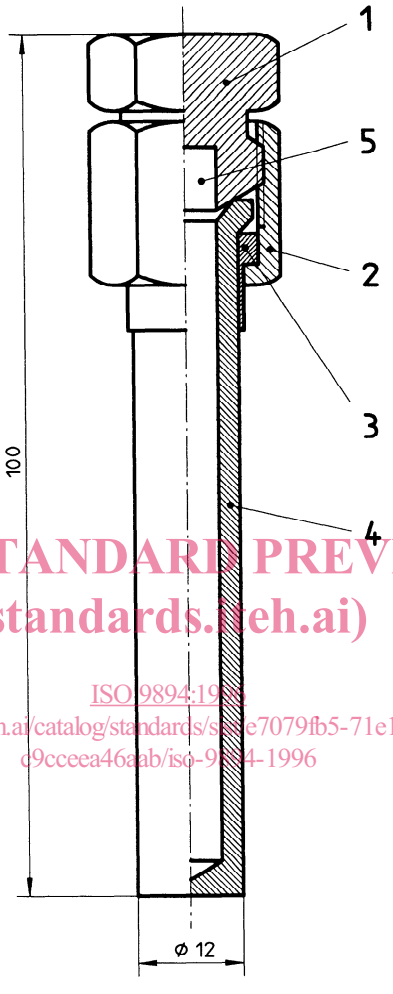


Figure 1 — Example of a PTFCE container (0,5 litre) for chemical analysis

Dimensions in millimetres

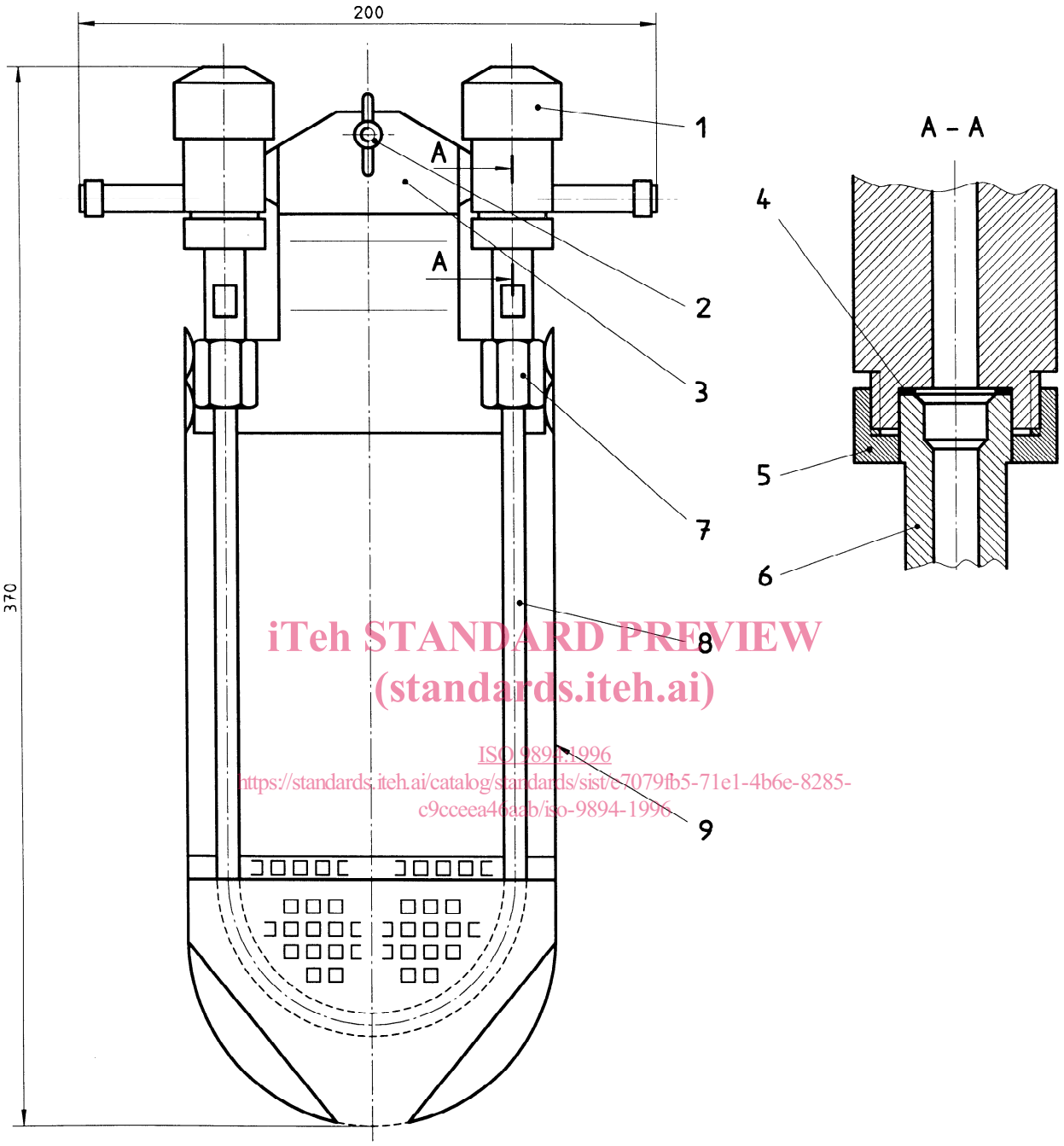


Key

1 Plug
2 Tightening nut
3 Centring nut
4 Sampling tube
5 Gasket (teflon)

Figure 2 — Example of PTFCE tube for uranium assay

Dimensions in millimetres



Key

1 Valve
2 Wing nut
3 Support
4 Gasket
5 Flare nut
6 Male coupling
7 Female coupling
8 Tube, length 500 mm
9 Protection cage

Figure 3 — Example of tube for isotopic analysis

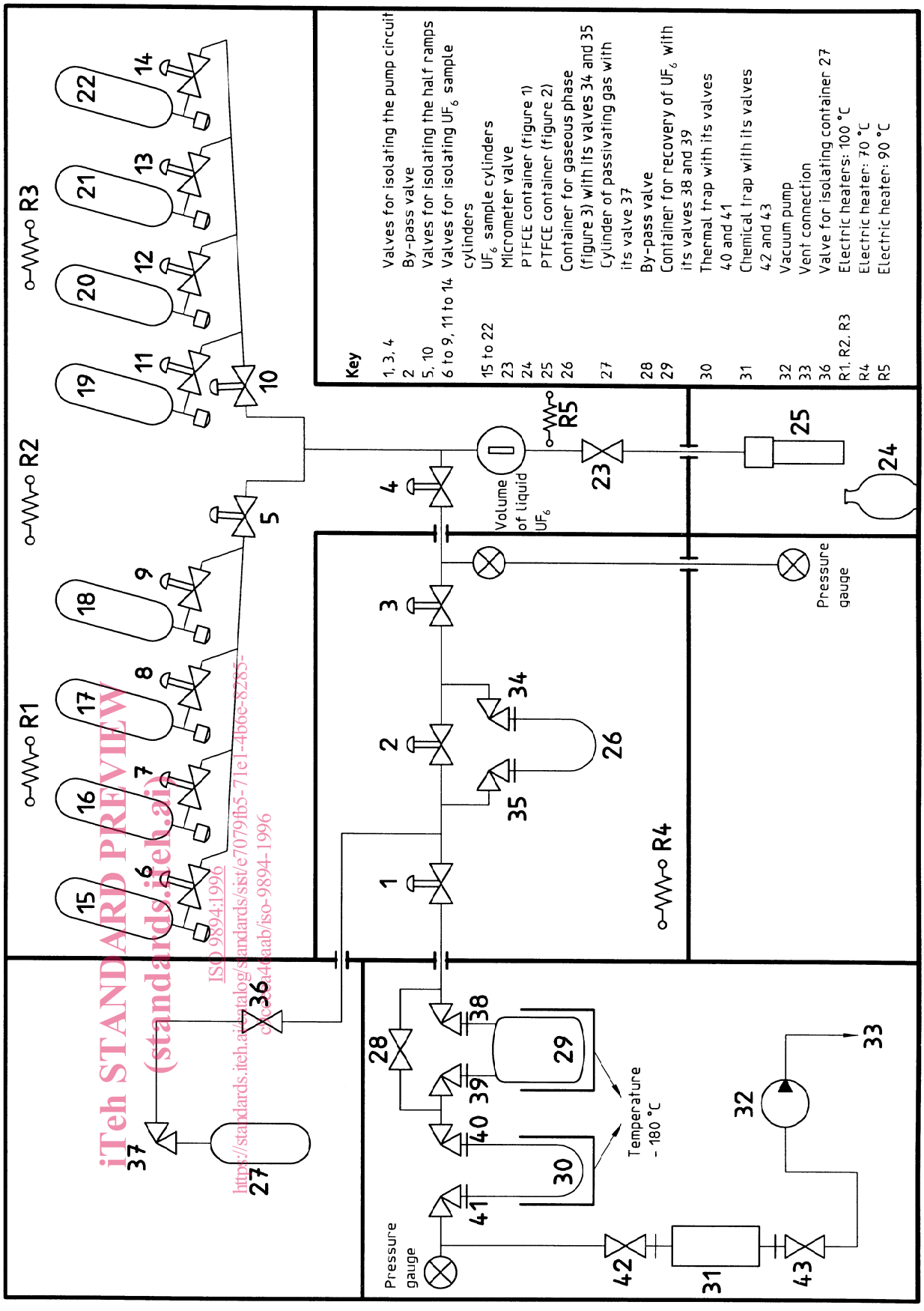


Figure 4 — Manifold for subsampling