# INTERNATIONAL STANDARD ISO 3978

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

# Aluminium and aluminium alloys — Determination of chromium — Spectrophotometric method using diphenylcarbazide, after extraction

*Aluminium et alliages d'aluminium — Dosage du chrome — Méthode spectrophotométrique à la diphénylcarbazide, après extraction*



**First edition — 1976-12-15**



**UDC 669.71 : 546.76 : 543.42** **Ref. No. ISO 3978-1976 (E)**

**Descriptors :** aluminium, aluminium alloys, chemical analysis, determination of content, chromium, spectrophotometric analysis, diphenylcarbazide.

Price based on 4 pages

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3978 was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*, and was circulated to the Member Bodies in November 1975.



It has been approved by the Member Bodies of the following countries :

| | | |
|---|---|---|
| Australia | India | Spain |
| Belgium | Italy | Turkey |
| Czechoslovakia | Japan | U.S.A. |
| Egypt, Arab Rep. of | Mexico | U.S.S.R. |
| France | Norway | Yugoslavia |
| Germany | Poland | |
| Hungary | South Africa, Rep. of | |

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Switzerland
United Kingdom



Printed in Switzerland

# Aluminium and aluminium alloys — Determination of chromium — Spectrophotometric method using diphenylcarbazide, after extraction

## 1 SCOPE

This International Standard specifies a spectrophotometric method using diphenylcarbazide, after extraction, for the determination of chromium in aluminium and aluminium alloys.

## 2 FIELD OF APPLICATION

This method is applicable to chromium contents between 0,002 and 0,60 % (*m/m*) of chromium (Cr).[1)]

## 3 PRINCIPLE

Dissolution of a test portion by a mixture of hydrochloric, nitric and sulphuric acids and evaporation to white $SO_3$ fumes. Taking up with water, filtration, if necessary, of the dehydrated silica and recovery of the chromium from the residue, after removal of the silica.

Oxidization of the Cr(III) to Cr(VI) by ammonium hexanitrocerate and extraction of the Cr with isobutylmethylketone. Passage of the Cr into an aqueous solution and formation of a coloured complex between the Cr(VI) and the diphenylcarbazide.

Spectrophotometric measurement of the coloured complex at a wavelength of about 545 nm.

## 4 REAGENTS

Throughout the analysis use only reagents of analytical grade and only distilled water or water of equivalent purity.

**4.1 Nitric acid,** $\rho$ about 1,40 g/ml, solution about 15 N.

**4.2 Sulphuric acid,** $\rho$ about 1,83 g/ml, solution about 36 N.

**4.3 Sulphuric acid,** $\rho$ about 1,23 g/ml, solution about 8 N.

**4.4 Hydrofluoric acid,** $\rho$ about 1,13 g/ml, 40 % (*m/m*) solution or about 23 N.

**4.5 Hydrochloric acid,** $\rho$ about 1,1 g/ml, 20 % (*m/m*) solution or about 6 N.

NOTE — During the analysis use this solution at a temperature of 5 or 10 °C. Store the solution in a refrigerator and, during the analysis, in an ice bath.

**4.6 Hydrochloric acid,** $\rho$ about 1,01 g/ml, 1,81 % (*m/m*) solution or about 0,5 N.

Dilute 84 ml of the hydrochloric acid solution (4.5) to 1 000 ml.

NOTE — Use this reagent cold, like the preceding reagent (4.5).

**4.7 Acide mixture**

Mix, in a convenient sized receptacle, 200 ml of hydrochloric acid, $\rho$ about 1,19 g/ml, solution about 12 N, 200 ml of the nitric acid solution (4.1) and 400 ml of water. Add cautiously, while shaking and cooling, 120 ml of the sulphuric acid solution (4.2). After cooling, make up the volume to 1 000 ml.

Store the mixture in a receptacle made of dark glass.

**4.8 Ammonium hexanitrocerate,** solution 0,04 N in Ce and 2 N in $H_2SO_4$.

Dissolve 2,19 g of ammonium hexanitrocerate ($(NH_4)_2[Ce(NO_3)_6]$) in a little water and add 25 ml of the sulphuric acid solution (4.3). Transfer the solution quantitatively to a 100 ml volumetric flask, make up to volume and mix.

**4.9 Isobutylmethylketone,** purified.

To a separating funnel of convenient size (for example 1 000 ml), transfer 250 ml of isobutylmethylketone [$CH_3COCH_2CH(CH_3)_2$] previously cooled to 5 to 10 °C, and add 250 ml of the hydrochloric acid solution (4.6) cooled to the same temperature. Shake vigorously for 1 min; then allow the two phases to separate. Run off the acid phase and discard it. Collect the isobutylmethylketone in a suitable receptacle.

NOTE — The reagent should be used cold.

1) This method may be extended to chromium contents of up to 1,5 % (*m/m*).