

Project: P-I247
Subject: Identity and analysis of chlorine
From: Secretariat
Date: 27 July 2010
Reference: P-I247/SPR/L026

Analysis of chlorine

For non-intermediate registrants, Annex VI of REACH requires analysis of the substance using spectral and chromatographic techniques.

The relevant portions of the annex read as follows:

Annex VI.

2.3.5. Spectral data (ultra-violet, infra-red, nuclear magnetic resonance or mass spectrum)

2.3.6. High-pressure liquid chromatogram, gas chromatogram

2.3.7. Description of the analytical methods or the appropriate bibliographic references for the identification of the substance and, where appropriate, for the identification of impurities and additives. This information shall be sufficient to allow the methods to be reproduced.

As chlorine is not an organic chemical and is a difficult substance to work with, some of the suggested techniques are not relevant or useful.

ECHA has suggested that irrelevant techniques should not be performed and appropriate techniques for qualitative and quantitative identification should be, but that this should be justified by waiving the suggested techniques and referencing scientific literature.

The Technical Committee has assessed the possible techniques and recommends that the following tests be performed to identify and quantify the substance:

Substance Identification

Raman spectroscopy – it is possible to take the Raman spectrum of chlorine, and this will provide information on the rotational and vibrational modes of the molecule. This information is specific for chlorine.

Mass spectrometry – the Electron Ionisation (EI) mass spectrum of chlorine will give a very clear fingerprint pattern, due to the isotopic composition of the chlorine nuclei. Major peaks should be observed at 70, 71 and 72 m/z, with the peak at 70 having a larger intensity than that at 71, which should be more intense than that at 72. Additional peaks at 35 and 36 should be observed. A sample spectrum can be found here:

<http://webbook.nist.gov/cgi/cbook.cgi?ID=C7782505&Units=SI&Mask=200#Mass-Spec>

Gas chromatography – Mass spectrometry – this test is technically possible, and also will identify the substance. Additionally, some quantification is possible by measuring the amounts of substance and carrier gas passing through the column.

UV-vis spectroscopy could be performed relatively easily, either by dissolving a sample of chlorine in CCl₄ or measuring the spectrum of a gaseous sample. This has been done and an example shown

in the attached report from Anwil. The resulting spectrum shows a characteristic band at 330 nm which can be used to identify chlorine.

IR spectroscopy is technically irrelevant, as chlorine provides no spectrum. ^1H and ^{13}C NMR spectroscopy should not be performed as they also will provide no signal. HPLC is technically not possible. These techniques will be waived.

Quantification

It was recommended that chlorine be quantified by titration, using the same method as that to quantify hypochlorite. A sample of chlorine is reacted in a solution of caustic soda, and the resulting products measured. This approach has been attempted by Anwil but could not however provide accurate results, due to the difficulty in dissolving and reacting a known amount of chlorine in a solution of caustic soda. However, each producing company has typical on-site methods for quantifying the amount of chlorine produced, and companies are advised to use these methods instead.

Report



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methods_Report_Anv

Waivers

Waivers for IR, ^1H and ^{13}C NMR and HPLC will be provided by Anwil.