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Spectra for the Identification of Monomers in Food Packaging

by

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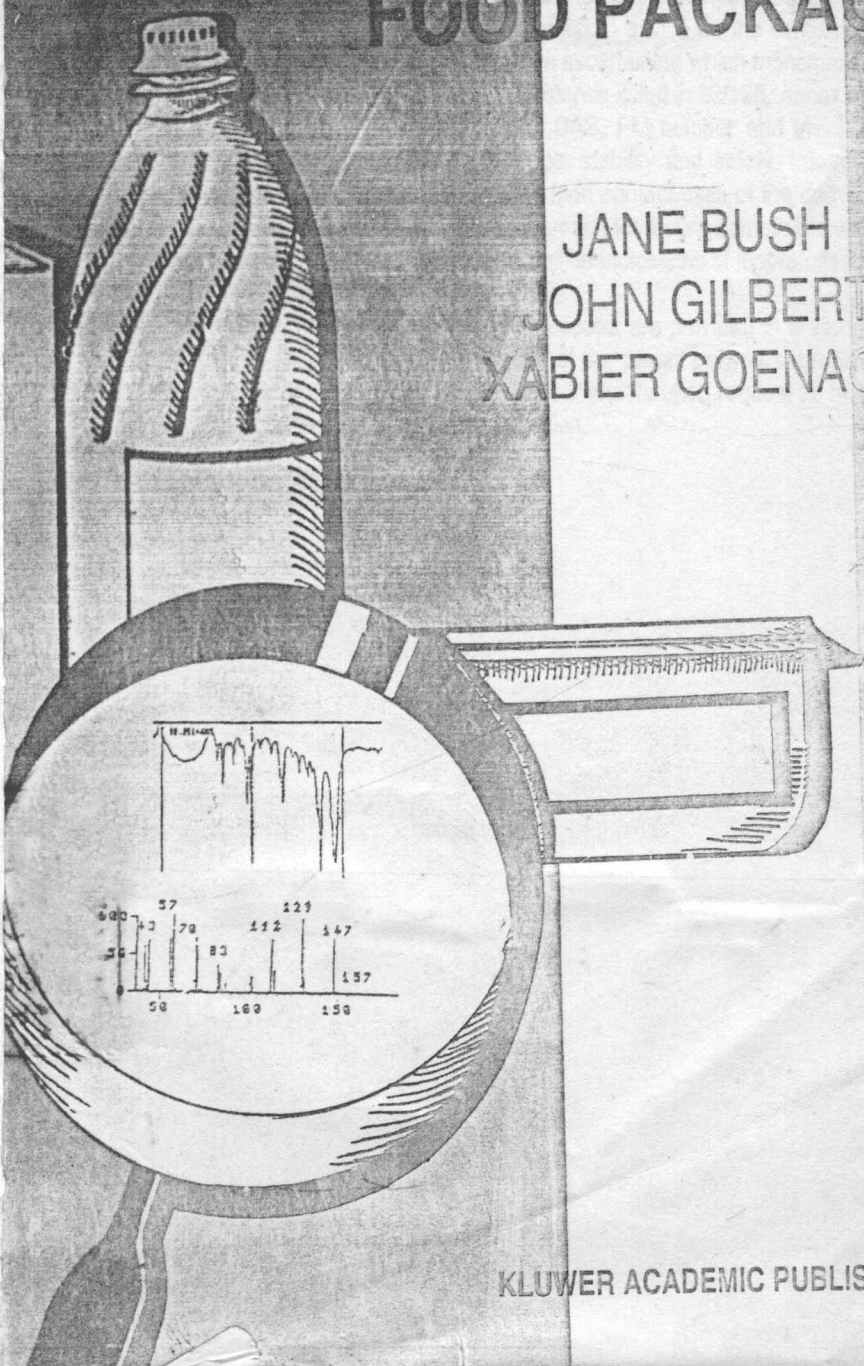
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SPECTRA FOR THE IDENTIFICATION OF MONOMERS IN FOOD PACKAGING

JANE BUSH, JOHN GILBERT & XABIER GOENAGA

This Handbook provides essential practical information for State Control Laboratories and for others concerned with ensuring compliance with European Community Directive 90/128 relating to plastics materials and articles intended to come into contact with foodstuffs.

The Handbook begins with a chapter giving practical advice on the sampling of retail packaging, determining the identity of the plastics and thus ascertaining which monomer residues are likely to be present. For every monomer or starting substance listed in 90/128, Annex A, there is an entry in the Handbook giving the structure, formulae, CAS, PM number and trivial name, together with information on physical characteristics, storage, stability and safety. Information is provided on known applications of the substances and the food contact uses of the derived plastics materials. There is a brief description of the analytical approach for testing compliance with SML or QM limits, and references to the published literature as well as indications of laboratories presently contracted by BCR to undertake method development.

Infrared spectra and electron ionization mass spectra are provided in a standard format for each substance and, in some cases, the IR spectra of the parent polymers are given. Most of the 166 substances listed in the Handbook will be made available upon request as reference standards as either the pure substance or as a calibrant solution.

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<u>Substance</u>	<u>CAS No.</u>	<u>PM/REF</u> <u>No.</u>	<u>Mol. wgt.</u>	<u>Page No.</u>
Abietic acid	00514-10-3	10030	302.46	11
Acetaldehyde	00075-07-0	10060	44.05	14
Acetic acid	00064-19-7	10090	60.05	17
Acetic acid, vinyl ester	00108-05-4	10120	86.09	21
Acetic anhydride	00108-24-7	10150	102.09	24
Acetylene	00074-86-2	10210	26.05	27
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Acrylic acid, ethyl ester	00140-88-5	11470	100.12	35
Acrylic acid, ethylene glycol monoester	00818-61-1	11830	116.12	38
Acrylic acid, isobutyl ester	00106-63-8	11590	128.17	41
Acrylic acid, isopropyl ester	00689-12-3	11680	114.15	44
Acrylic acid, methyl ester	00096-33-3	11710	86.09	45
Acrylic acid, n-butyl ester	00141-32-2	10780	128.17	48
Acrylic acid, propyl ester	00925-60-0	11980	114.15	51
Acrylic acid, sec butyl ester	02998-08-5	10810	128.17	52
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Acrylonitrile	00107-13-1	12100	53.06	56
Adipic acid	00124-04-9	12130	146.14	60
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Albumin, coagulated with formaldehyde	-	12340	-	no entry
Alcohols, aliphatic, monohydric, saturated, linear, primary (C4-C22)	-	12370	-	no entry
11-Aminoundecanoic acid	02432-99-7	12788	201.31	64
Azelaic acid	00123-99-9	12820	188.23	66
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2,2-Bis(4-hydroxyphenyl) propane bis(2,3-epoxypropyl) ether	01675-54-3	13510	340.42	84
2,2-Bis(4-hydroxyphenyl) propane, bis(phthalic anhydride)	38103-06-9	13530	530.00	88
3,3-Bis(3-methyl-4-hydroxyphenyl)-2-indolinone	47465-97-4	13600	345.40	89
Butadiene	00106-99-0	13630	54.09	90
1,3-Butanediol	00107-88-0	13690	90.12	93
1-Butanol	00071-36-3	13840	74.12	96
1-Butene	00106-98-9	13870	56.11	99
2-Butene	00107-01-7	13900	56.11	100
Butyraldehyde	00123-72-8	14110	72.11	101
Butyric acid	00107-92-6	14140	88.11	104
Butyric anhydride	00106-31-0	14170	158.20	107
Caprolactam	00105-60-2	14200	113.16	110
Caprolactam, sodium salt	02123-24-2	14230	135.14	no entry

<u>Substance</u>	<u>CAS No.</u>	<u>PM/REF No.</u>	<u>Mol. wgt.</u>	<u>Page No.</u>
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Cellulose	09004-34-6	14500	-	120
Chlorine	07782-50-5	14530	70.91	122
Citric acid	00077-92-9	14680	192.13	123
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o-Cresol	00095-48-7	14740	108.14	128
p-Cresol	00106-44-5	14770	108.14	131
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1,4-Diaminobutane	00110-60-1	15250	88.15	140
Dicyclohexylmethane-4,4'- -diisocyanate	05124-30-1	15700	262.35	143
Diethyleneglycol	00111-46-6	15760	106.12	147
1,2-Dihydroxybenzene	00120-80-9	15880	110.11	151
1,3-Dihydroxybenzene	00108-46-3	15910	110.11	155
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4,4'-Dihydroxybenzophenone	00611-99-4	15970	214.22	162
4,4'-Dihydroxybiphenyl	00092-88-6	16000	186.21	165
Dimethylaminoethanol	00108-01-0	16150	89.14	168
3,3'-Dimethyl-4,4'- diisocyanatobiphenyl	00091-97-4	16240	264.29	171
Dipentaerythritol	00126-58-9	16480	254.28	172
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Diphenylmethane-2,4'- diisocyanate	05873-54-1	16600	250.26	176
Diphenylmethane-4,4'- diisocyanate	00101-68-8	16630	250.26	180
Dipropyleneglycol	00110-98-5	16660	134.18	184
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Ethylene	00074-85-1	16950	28.05	195
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Ethyleneglycol	00107-21-1	16990	62.07	200
Ethyleneimine	00151-56-4	17005	43.07	204
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1-Hexadecanol	36653-82-4	18310	242.45	220
Hexamethylenediamine	00124-09-4	18460	116.21	223
Hexamethylene diisocyanate	00822-06-0	13640	168.20	227
Hexamethylenetetramine	00100-97-0	18670	140.19	231
p-Hydroxybenzoic acid	00099-96-7	18880	138.12	235

<u>Substance</u>	<u>CAS No.</u>	<u>PM/REF</u> <u>No.</u>	<u>Mol. wgt.</u>	<u>Page No.</u>
Isobutene	00115-11-7	19000	56.11	238
Lauric acid	00143-07-7	19470	200.32	239
Lignocellulose	11132-73-3	19510	-	no entry
Maleic acid	00110-16-7	19540	116.07	243
Maleic anhydride	00108-31-6	19960	98.06	245
Methacrylic acid	00079-41-4	20020	86.09	248
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Methacrylic acid, ethyl ester	00097-63-2	20890	114.15	254
Methacrylic acid, ethylene glycol monoester	00868-77-9	21190	130.14	257
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Methacrylic acid, isopropyl ester	04655-34-9	21100	128.17	263
Methacrylic acid, methyl ester	00080-62-6	21130	100.12	264
Methacrylic acid, propyl ester	02210-28-8	21340	128.17	267
Methacrylic acid, sec-butyl ester	02998-18-7	20140	142.20	268
Methacrylic acid, tert-butyl ester	00585-07-9	20170	142.20	269
Methacrylic anhydride	00760-93-0	21460	154.17	270
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1,5-Naphthalene diisocyanate	03173-72-6	22420	210.19	288
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1-Nonanol	00143-08-8	22480	144.26	293
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1-Octanol	00111-87-5	22600	130.23	300
1-Octene	00111-66-0	22660	112.22	303
Palmitic acid	00057-10-3	22780	256.43	307
Pentaerythritol	00115-77-5	22840	136.15	311
1-Pentanol	00071-41-0	22870	88.15	314
Phenol	00108-95-2	22960	94.11	317
1,3-Phenylenediamine	00108-45-2	23050	108.14	320
Phenyl isocyanate	00103-71-9	23125	119.12	323
Phosphoric acid	07664-38-2	23170	98.00	327
Phthalic acid, diallyl ester	00131-17-9	23230	246.27	329
Phthalic anhydride	00085-44-9	23380	148.12	332
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beta-Pinene	00127-91-3	23500	136.23	336
Polyethyleneglycol	25322-68-3	23590	-	339
Polypropyleneglycol	25322-69-4	23650	-	340
1,2-Propanediol	00057-55-6	23740	76.12	342
1-Propanol	00071-23-8	23800	60.10	345
2-Propanol	00067-63-0	23830	60.10	348
Propionaldehyde	00123-38-6	23860	58.08	351
Propionic acid	00079-09-4	23890	74.08	352
Propionic anhydride	00123-62-6	23950	130.14	356
Propylene	00115-07-1	23980	42.08	359
Propylene oxide	00075-56-9	24010	58.08	360
Resin acids and rosin acids	73138-82-6	24070	-	362

<u>Substance</u>	<u>CAS No.</u>	<u>PM/REF</u> <u>No.</u>	<u>Mol. wgt.</u>	<u>Page No.</u>
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Rosin tall oil	08052-10-6	24160	-	366
Rosin wood	09014-63-5	24190	-	no entry
Rubber, natural	09006-04-6	24250	-	368
Sebacic acid	00111-20-6	24280	202.25	369
Sorbitol	00050-70-4	24490	182.17	373
Soybean oil	08001-22-7	24520	-	375
Stearic acid	00057-11-4	24550	284.49	377
Styrene	00100-42-5	24610	104.12	381
Succinic acid	00110-15-6	24820	118.09	385
Sucrose	00057-50-1	24880	342.30	388
Terephthalic acid	00100-21-0	24910	166.13	390
Terephthalic acid, dimethyl ester	00120-61-6	24970	194.19	394
Tetrahydrofuran	00109-99-9	25150	72.11	397
Tetraethyleneglycol	00112-60-7	25090	194.23	400
N.N.N.'N'-Tetrakis(2-hydroxypropyl)ethylenediamine	00102-60-3	25180	292.42	403
2,4-Toluene diisocyanate	00584-84-9	25210	174.16	404
2,6-Toluene diisocyanate	00091-08-7	25240	174.16	408
2,4-Toluene diisocyanate, dimer	26747-90-0	25270	348.32	412
Trialkyl (C5-C15)acetic acid, 2,3-epoxypropyl ester	-	25360	-	no entry
2,4,6-Triamino-1,3,5-triazine	00108-78-1	25420	126.12	415
Triethyleneglycol	00112-27-6	25510	150.18	419
1,1,1-Trimethylol propane	00077-99-6	25600	134.18	422
Tripropyleneglycol	24800-44-0	25910	192.26	425
Urea	00057-13-6	25960	60.06	428
vinyl chloride	00075-01-4	26050	62.50	430
Vinylidene chloride	00075-35-4	26110	96.94	433

CHAPTER 1

Legal Framework

In the European Community one of the main controls on materials and articles for food contact use is through Directive 90/128/EEC (1) which restricts the range of monomers and other starting substances that can be used for the production of plastics. The control is through the use of a positive list of authorised substances grouped into section A of approved substances and section B of substances with provisional approval pending a decision on inclusion in section A. Directive 90/128 together with subsequent amendments listed 163 substances in section A, although it was envisaged that the situation would never be static and there would always be movements from section B to section A as well as approval and adoption of new substances. In addition to positive list controls the Directive places limits on certain substances in the form of maximum levels of residual substance permitted in the finished plastic (QM limit) and/or maximum amount of substance permitted to migrate into foods or food simulants under defined conditions (SML limit).

Implementation of legislation

Although the principles that are intended to govern the control of materials and articles are clear from the Directive, the practical problems of implementation or the development of approaches that should be adopted by enforcement authorities in real situations have not yet been addressed. The most systematic approach to control has been elaborated in the Netherlands (2) to meet Dutch Regulations in existence before EC 90/128. The approach used has been to initially identify polymeric materials by infra-red spectroscopy and then to identify substances in solvent extracts by gas chromatography/mass spectrometry and liquid chromatography.

Practical applications of this approach over many years has shown that considerable experience is required in knowing what polymer is used in what food contact situation, as well as the likely additives and other constituents that might be anticipated. Spectroscopic and chromatographic data-base information as well as access to authentic reference standards is essential to carry out this enforcement work.

In 1990, the UK Ministry of Agriculture, Fisheries and Food funded a project with Maurice Palmer Associates in Cambridge (UK) aimed at collecting monomers and other starting substances from industry to form a reference collection for use by enforcement authorities. It was found that often it was difficult to identify a source of such substances. Further support was given in 1991 by the Community Bureau of Reference (BCR) of the Commission of European Communities for the completion of the reference collection, the preparation of a database of physicochemical data and a handbook of spectra of monomers and starting substances. This Handbook is seen as being primarily of practical assistance to enforcement laboratories in the European Community but may well also be useful to industry, Universities and other research establishments. The book is envisaged as being a reference source of information that can be used by relatively inexperienced laboratories to develop expertise in the area of food contact materials. In addition to the spectroscopic and other information contained in the Handbook, reasonable requests for the substances themselves will be met free of charge on application to the address below. The substances will be supplied as reference standards (1 g) or as reference solutions where appropriate from:-

Plastics Reference Collection
Ministry of Agriculture, Fisheries and Food,
Food Science Laboratory,
Norwich Research Park,
Colney,
Norwich, NR4 7UQ (U.K.)

Scope of the Handbook.

Altogether 106 of the 163 substances listed in section A have been obtained as commercially available samples either from industry or from laboratory chemical suppliers. Certain substances which were natural materials of ill-defined composition such as rubber, cellulose and albumin were not included in the collection although they are listed in the Handbook. Volatile monomers and gases (17 in total) which could not be easily retained in pure form were prepared in solution at a defined concentration, the solvent being chosen on the basis of that most likely to be suitable for the purposes of analysis. It was appreciated that with the constant amendments to Directive 90/128 EEC no Handbook could ever be entirely complete or up-to-date and this must be appreciated as an inevitable limitation. An extension to this Handbook will be needed to include those additional monomers and starting substances which will have been granted approval for use.

Entries for each substance give the structural formula, the CAS number and the PM reference number which is the number by which substances are listed in Directive 90/128. Alternative names whether systematic IUPAC nomenclature or trivial names are given as a further aid to identification. Physical characteristics such as solubility in common organic solvents are described. Some indications for handling are given, but safety requirements vary from country to country and therefore indications of safety given are very general and not intended to have any legal status. For example, in the UK safe handling of chemicals is regulated by the Control of Substances Hazardous to Health Regulations (COSHH) and the indications in this Handbook are not meant to surpass the requirements of making a full COSHH assessment. Information provided on stability is intended as advisory, being based on that provided in good faith by suppliers or from published sources, and cannot be taken as definitive.

Information on current uses of monomers and food contact applications, whilst invaluable to the enforcement laboratory, is often anecdotal and thus difficult to assemble in any systematic way. There is also the added complication that whilst information particularly on applications may be relevant for one country there may be quite different practice in other countries. To cope with these difficulties entries on usage and applications for each substance have been circulated widely for comment in the UK, The Netherlands, Germany, France and Spain as well as to the Association of Polymer Manufacturers Europe (APME).

The infra-red spectra are intended to complement the already available library collections of Hummel and Scholl (3) and Sadtler (4). Spectra are given for substances already in these libraries as well as for substances for which spectra have not been previously published. All spectra have been run on more than one instrument as a check on the authenticity of the data and frequently in a number of different formats. In a small number of instances where inconsistencies were found in spectra either between those for the Handbook and other library spectra or between those obtained on two different instruments, unless the differences could be explained on interpretation then additional samples were obtained from a second source. As the infra-red spectra were envisaged as likely to be used primarily as an aid for identification of polymers, in a number of instances for major polymers the spectra of the polymers are also given.

Mass spectra are similarly meant to complement existing data bases, and have also been obtained on two or more instruments in order to control and cross-check the quality of spectra ultimately adopted for the Handbook. In some instances through lack of volatility or for other reasons mass spectra could not be obtained - where this was the case it is indicated, although where suitable spectra could be obtained of volatile derivatives, then this approach was adopted. Gas chromatographic retention indices are provided as an additional aid to assist in identification of monomers.

The Handbook also contains a brief entry on analytical methods which gives an outline of the approach to be used for measuring to test for compliance with QM or SML limits. Other bodies are actively engaged on method development and validation of analytical methods in support of 90/128 EEC, in particular the European Committee for Standardisation (CEN). Where it was known that a CEN method was in development this has been indicated together with a note of the organisation responsible for the method development. In order to meet the urgent need for more methods, the BCR Programme has undertaken a project for the development of methods for more than 30 substances with restrictions in 90/128/EEC. When ready these methods will be normalised by CEN. The Council of Europe has also been active in the area of assessment of published methods for the analysis of monomers and other starting substances, and ILSI Europe has developed an analytical methods data-base that gives a comprehensive coverage of published analytical procedures (5). In this Handbook we have cited what we believe to be the most relevant references to analytical methods in the published literature taking account of the likely availability of the technique employed and the relevance of the matrices tested.

It is intended that this Handbook should be used as a general reference source to assist in the identification of plastics materials and articles obtained for enforcement purposes. Infra-red and mass spectra for unknown materials can be compared with the reference spectra in the Handbook for identification and where appropriate the references to further analytical methods can be pursued.

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3552 AS Utrecht (Netherlands)

The success in producing this Handbook has also been due to the assistance in supplying samples and to the information made freely available from industrial and Government sources too numerous to mention. We are especially grateful to Mr R. Ashby (ICI, Wilton), Dr R. Franz (Fraunhofer Institute, Munich), Dr P.Tice (PIRA, Leatherhead), Mr R. Rijk (TNO, Zeist), Dr L. Rossi (EC, Brussels) and Mr D Shorten (BP Chemicals, UK) for their advice and comments on entries to the Handbook.

References

- (1) Commission Directive of 23 Feb 1990 relating to plastics materials and articles intended to come into contact with foodstuffs (90/128/EEC). Official Journal of the European Communities No L 349/26, 1990.
- (2) van Battum, D., and van Lierop, J.B.H., 1988, Food Additives and Contaminants, 5, 381-395.

(3) D.O. Hummell and F. Scholl, Atlas of polymer and plastics analysis. Vol 1-3. Hanser publishers (Munich, Vienna, New York and Barcelona) and VCH (Weinheim, New York, Basel and Cambridge).

(4) Sadtler Monomers and Polymers Library 10600 spectra. Heyden & Son Ltd. (London).

(5) ILSI Data base of Analytical methods (ILSI Europe, Avenue E Mounier 83, Brussels)

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Selection of entries

The monomers selected for entry are those in list A of directive 90/128/EEC that have SML or QM restrictions. Since it is the intention to update this directive annually, with additions, deletions, and movement of substances from list B to list A, readers should check the most recent amendment to establish the exact status of a substance at the time of enquiry.

Some substances listed in the directive could not be traced and were not available either from industry or from the normal chemical supply companies. In these cases therefore, no reference substance is available. Further, in some cases it was not possible to attribute a final use to some of these substances. In these cases, no entry has been included in the atlas except for a listing in the index.

Literature references

The selection criteria for references to analytical methods were that the methods should relate to foods, simulants or food contact materials. At the time of writing, analytical methods are under development for all substances with restrictions, under the auspices of CEN (Working Group 5 of Technical Committee 194) and the BCR (DGXII/C/5, Measurements and Testing Programme 1992). It is the intention of DGIII/C/1 to provide a compendium of analytical methods as part of the general "Notes for guidance" that will complement directives. This compendium will include methods that are finalised as well as a brief description of the basis of methods under development. The current draft of this compendium document is entitled "Guidelines for the compliance with and enforcement of community directives on