

Feature Article

Acrylamide Determination in Food

Standards and Derivatization Reagents

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Introducing New Products and Applications At Sigma-Aldrich, we strive continually to add new and relevant products to help the challenging tasks facing analytical chemists.



Picture Ingo Haag, PhD
Analytical Reagents / Chromatography
Marketing Manager

Dear Colleague,

Spring is a time of change, and this spring is no exception. We are pleased to produce the fruits of our winter labors: the simultaneous release of both Supelco and Fluka/Riedel-de Haën catalogs. For the first time, catalogs from Sigma-Aldrich's analytical brands will be combined in a specially designed blue container: the Analytical Box. Serving as a "one stop shop," the Analytical Box provides convenient, fast and easy sourcing and ordering of thousands of analytical standards, reagents and chromatography consumables, including Supelco brand HPLC and GC columns and CHROMASOLV® LC-MS solvents, to name only a few. The Analytical Box (or "A-Box," as we've been affectionately calling it) is the most comprehensive source of products for analytical chemistry laboratories. Call us or go to the link below to receive your A-Box.

Besides the news about the release of the Analytical Box, throughout this issue of Analytix you'll see other articles and announcements about new and featured products for many areas of current interest in analytical chemistry. Two articles in particular testify to the importance of analytical chemistry to monitor the quality and safety of our food supply: amine compounds in fish and acrylamide in cooked foods. Sigma-Aldrich offers convenient kits, standards and separation and purification devices for measuring these troublesome compounds.

Finally, as part of our ongoing effort to share our knowledge and expertise, please check out the BRC card for a list of upcoming Sigma-Aldrich seminars in your area.

What can we do for you?

If you would like to find out whether the combined analytical expertise of scientists in our Fluka, Riedel-de Haën and Supelco brands can help solve your most challenging analytical problem, please contact our Technical Service Team using the contact data on the back page. Our chemists enjoy being challenged by your questions!

Kind regards,

Ingo Haag, PhD
Analytical Reagents / Chromatography
Marketing Manager
ihaag@europe.sial.com

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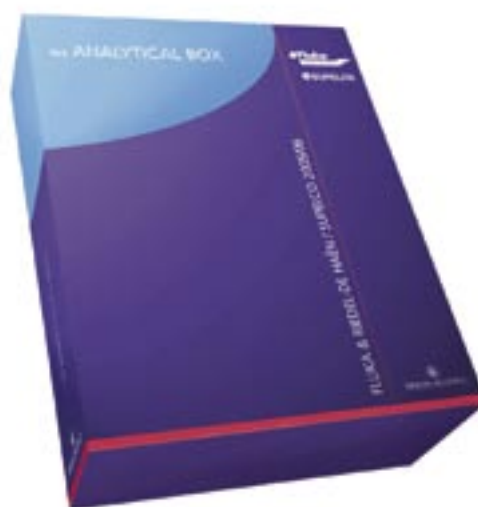
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Acrylamide in food: Kits, columns and tubes for sensitive GC-MS and LC-MS analyses and solid phase extraction Acrylamide is a commonly analyzed food-borne carcinogen. Sigma-Aldrich has convenient kits, LC and GC columns and sample prep methods for fast and reliable analysis of acrylamide by both analytical techniques.

By Rainer Walz, PhD, Product Manager, Fluka/Riedel-de Haën ...rwalz@sial.com
and An Trinh, Product Manager, Supelcoatrn@sial.com

Formation and health risks of acrylamide in food

Acrylamide analysis has been a very hot topic since the chemical was identified in food in 2002 by researchers at the Swedish National Food Administration.¹ Since then, alarmingly high concentrations of acrylamide have been found in many popular processed foods including French fries, potato chips, breakfast cereals, coffee, chocolate, peanut butter, crisp bread and pastries. Researchers at the Department of Environmental Chemistry, Stockholm University, measured moderate levels of acrylamide (5-50 µg/kg) in heated protein-rich foods and higher levels (150-4,000 µg/kg) in carbohydrate-rich foods, such as potatoes and bread. Acrylamide could not be detected in unheated control or boiled foods (<5 µg/kg). Based on average consumption levels, consuming certain heated foods could lead to a daily intake of a few tens of micrograms of acrylamide.²

Acrylamide ends up in food via two routes: as a leachate from packaging materials and as an unintentional consequence of the cooking process when high carbohydrate, low protein foods are fried, roasted or baked at high temperatures. A proposed mechanism of its formation is the reaction of asparagine and a carbonyl-containing compound. The mechanism involves formation of a Schiff base followed by decarboxylation and elimination of either ammonia or a substituted imine to yield acrylamide.³ Temperature plays an important role in the reaction.

In February 2005, the Joint FAO/WHO Expert Committee on Food Additives and Contaminants (JECFA) met to consider the possible health risks associated with acrylamide and other food contaminants.⁴ Although early reports suggested that acrylamide is a neurotoxin, reproductive toxin and teratogen, the Committee concluded that cancer is the most important toxic effect of acrylamide and that consumption of foods with acrylamide at levels currently found in some processed foods poses a public health concern. The low threshold of toxicity coupled with the high degree of activity surrounding acrylamide research necessitates reliable, fast and sensitive analytical methods.

Analytical methods to measure acrylamide in food

A review of the scientific literature shows that many different analytical approaches have been applied to the analysis of acrylamide in food, with GC-MS and LC-MS accounting for the majority of the citations. Analysis of acrylamide in food is difficult because of the high level of endogenous matrix interferences inherent to starchy foods. Sample clean-up is necessary to prevent starches and oils from interfering with GC-MS or LC-MS analyses.

Table 1 Acrylamide Kit and components

| Cat. No. | Brand | Description* | Package Size |
|----------|-------|---|--------------|
| 72615 | Fluka | Acrylamide Kit (for 12 determinations according to the Grob method) | 1 Kit |

*Contents of Acrylamide Kit (Cat. No. 72615):

| Product | Description | Package Size and Type |
|---|---------------------------------------|-----------------------|
| Acrylamide Standard Solution | 500ppm in acetonitrile | 5mL (Certan® vial) |
| Acrylamide-D ₃ Standard Solution | 500ppm in acetonitrile | 5mL (Certan® vial) |
| Methacrylamide Standard Solution | 500ppm in acetonitrile | 5mL (Certan® vial) |
| Butyramide Standard Solution | 25ppm in acetonitrile | 5mL (Certan® vial) |
| 1-Propanol | puriss., p.a., >99.5% (GC) | 250mL glass bottle |
| n-Hexane | puriss., >99.0% (GC) | 100mL glass bottle |
| Acetonitrile | For residue analysis, >99.9% (GC) | 50mL glass bottle |
| Oil | Suitable for acrylamide determination | 50mL glass bottle |

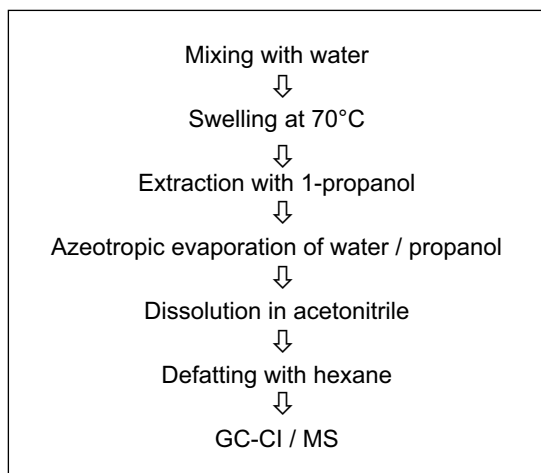


Figure 1 Summary of Grob GC-Cl/MS method

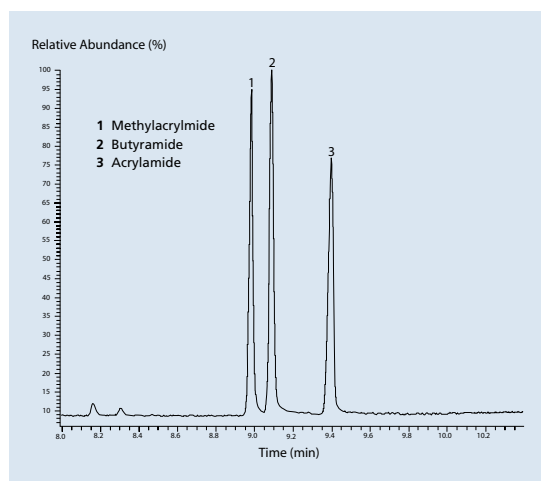


Figure 2 GC-Cl/MS chromatogram of methacrylamide, butyramide and acrylamide. (Column: SUPELCOWAX™ 10, 30m x 0.25mm ID x 0.25µm df. Oven: Gradient starting at 70°C (1 min.) to 220°C at 15° C/min, hold 2 min. Injection volume: 1µL. Carrier gas: He, 20 cm/sec. Injection: on column.)

Food analysis GC-Cl/MS method

A common GC-MS technique for acrylamide analysis in food is based on a method developed by Grob, et al.⁵ The sample preparation involves several steps (Figure 1): swelling the sample in water, centrifugation, addition of small amount of oil, evaporation, reconstitution in acetonitrile and removal of fats and lipids by hexane extraction. Internal standards are added both before and after extraction. The acetonitrile extract containing the acrylamide is analyzed by GC-MS on a Carbowax 20M capillary column with positive ion chemical ionization mass spectroscopic detection. Supelco SUPELCOWAX™ 10 capillary columns are ideally suited for the separation and MS detection.

The Grob method requires several high quality reagents and standards that initially were not available from a single supplier. To address this, scientists at Sigma-Aldrich's Fluka and Riedel-de Haën divisions developed a ready-to-use kit that makes acrylamide determination faster and easier. The Acrylamide Kit (Table 1) contains all the necessary standards and solvents for twelve determinations of acrylamide using the Grob method. The concentrations and specifications of the acrylamide, D₃-acrylamide and methacrylamide standard solutions were designed specifically for this method. Each Kit also contains all sample preparation protocols and the GC test chromatogram and MS spectra of the standards (Figure 2). More information on the Acrylamide Kit can be found in the Product Listing (Table 2) and on the web site:

www.sigma-aldrich.com/kit_gc_acrylamide

Table 2 Product List: Solvents, SPE Tubes and HPLC and GC Columns for acrylamide detection

| Cat. No. | Brand | Description | Packaging Size |
|----------|----------------|---|----------------|
| 24079 | Supelco | SUPELCOWAX™ 10 Capillary GC column | 1 Unit |
| 52784-U | Supelco | Discovery DSC-MCAX SPE tubes 3mL, 300mg bed weight | Pack of 54 |
| 52606-U | Supelco | Discovery DSC-C18 SPE tubes 6mL, 1gram bed weight | Pack of 30 |
| 57020-U | Supelco | SPE Tube Adapter for 1, 3 & 6mL tubes | Pack of 12 |
| 567507-U | Supelco | Discovery F5 HPLC column, 15cm x 4.6mm, 3µm particles | 1 Unit |
| 34967 | Riedel-de Haën | Acetonitrile (LC-MS CHROMASOLV®) | 1L, 2.5L |
| 39253 | Riedel-de Haën | Water (LC-MS CHROMASOLV®) | 1L |

SPE-LC-ESI/MS method

Although the Grob GC-MS method gives excellent results and is widely accepted, the extensive sample preparation may be a drawback for some analysts, especially those who prefer LC methods. In the SPE-LC-MS method developed by researchers at Sigma-Aldrich's Supelco division, a mixed-mode cation-exchange SPE phase (Discovery DSC-MCAX) in series with a C18 SPE phase (Discovery DSC-18) is used to extract acrylamide from food prior to LC-MS analysis (Figure 3). The two SPE tubes are connected in series via an SPE tube adapter. The MCAX cartridge has both hydrophobic and ionic interactions and retains starch and oily interferences. The C18 cartridge retains the acrylamide by a weak hydrophobic mechanism. The eluate from the C18 cartridge is analyzed by LC-MS using a fluorinated reversed phase column, Discovery F5. Unlike standard C18 HPLC phases, the F5 column gives enhanced retention of the polar acrylamide and excellent retention and peak shape using a completely aqueous mobile phase (Figure 4). Complete details on the SPE-LC-ESI/MS method is described in the Supelco Reporter, Volume 22.5 (October 2004) which is available by contacting us or by visiting the website:

www.sigma-aldrich.com/spe_acrylamide

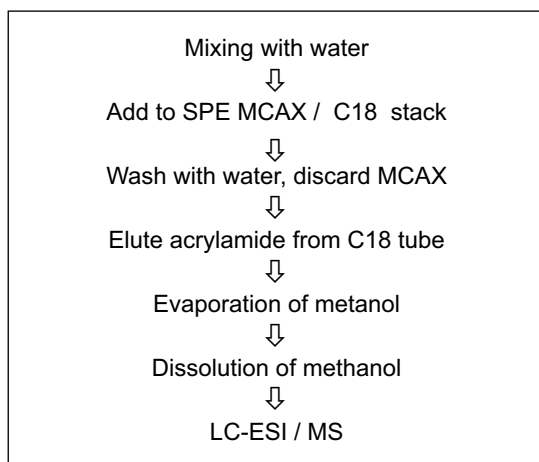


Figure 3 Summary of SPE-LC-ESI/MS method

Conclusions

Sigma-Aldrich offers the complete solution for acrylamide analysis, no matter which analytical method you prefer. For GC-MS, we offer the Acrylamide Kit and SUPELCOWAX™ 10 GC capillary columns. By choosing the Kit, time consuming standard preparation is avoided and detailed protocols take away any method development time. For LC-MS, the Discovery line of SPE and HPLC phases provides the necessary retention, efficiency and selectivity. This method requires no derivatization to produce sufficient retention and detection, and sample prep is fast and easy with high recoveries of acrylamide from complex matrices.

An excellent resource for up-to-date information on acrylamide research and regulations is the website www.acrylamide-food.org. The site is maintained jointly by the FAO (UN), JIFSAN (US FDA and University of Maryland) and the WHO's Food Safety Department.

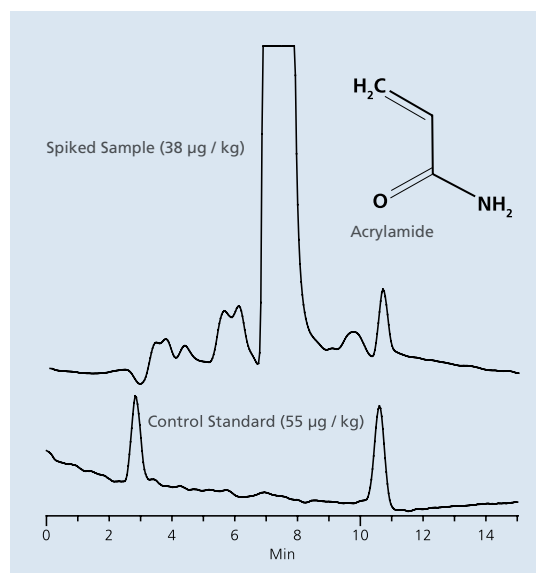


Figure 4 LC-ESI/MS chromatogram of control and acrylamide-spiked potato chip extract. (Column: Discovery F5, 15cm x 4.6mm, 3µm. Mobile phase: 100% Water (LC-MS CHROMASOLV™). 0.3mL/min, 35°C. Injection volume: 5µL. Standard: 48µg/mL. MS conditions: +ESI, SIM 72 m/z.)

References

- [1] Swedish National Food Administration: Information about acrylamide in food. www.slv.se/Download/Document/approvedDocs/enginformatiakrvl.htm, 24 April 2002.
- [2] E. Tareke, P. Rydberg, P. Karlsson, S. Eriksson, M. Tornqvist; J. Agric. Food Chem. 2002; 50(17); 4998-5006.
- [3] D. Zyzak, R. Sanders, M. Stojanovic, D. Tallmadge, B. Eberhart, D. Ewald, D. Gruber, T. Morsch, M. Strothers, G. Rizzi, M. Villagran; J. Agric. Food Chem. 2003; 51(16); 4782-7.
- [4] Joint FAO/WHO Expert Committee on Food Additives, Sixty-fourth meeting, Rome, 8-17 February 2005 (http://www.who.int/entity/ipcs/food/jecfa/summaries/en/summary_report_64_final.pdf).
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Improved analytical methods to determine the freshness of seafood

Sigma-Aldrich offers kits, standards and procedures for measuring TVB, TMAO and biogenic amines in fish tissue can be detected at visible wavelengths and at picomole concentrations.

By Rainer Walz, PhD, Product Manager, Fluka/Riedel-de Haën rwalz@sial.com

One of the most important applications of analytical chemistry is for the assessment of food quality, safety and nutritional value. Within the food industry, there are thousands of methods to determine levels of specific compounds that are indicators of some quality aspect of the food in question. Many of these tests are dictated by regulatory agencies. The focus of this article is the measurement of compounds that are indicators of the freshness of seafood products.

A challenge to the fisheries industry is how to maintain freshness of the catch over the distance between the catch site and the cannery or processing plant, and until it reaches the consumer. Seafood has a notoriously short freshness span between catch and processing; freshly caught fish that isn't quickly processed or iced rapidly develops an unpleasant "fishy" odor. (Interestingly, fresh fish has little or no odor.) This unpleasant odor arises from a variety of compounds, but mostly from amines that are formed from the breakdown of amino acids in the fish tissue. Obviously, spoiled seafood has very little commercial value, so having reliable methods for the determination of freshness and spoilage is of great importance to both producers and consumers. There are three groups of compounds that are of particular interest from an analytical standpoint as freshness indicators: total volatile bases (TVB), trimethylamine N-oxide (TMAO) and the biogenic amines index (BAI).



"Seafood has a notoriously short freshness span between catch and processing"

Total Volatile Bases (TVB)

Regulations in the EU (95 / 149 / EC) specify a maximum value for TVB in different fish species. The analytical method described in 95 / 149 / EC is applicable to TVB concentrations from 5 mg / 100 g up to 100 mg / 100 g. The volatile nitrogenous bases are extracted from the fish sample using 0.6 M perchloric acid. After neutralization with alkali the extract undergoes steam distillation. An acid receiver absorbs the volatile basic compounds in the extracted sample. The TVB concentration is determined by titration of the absorbed bases.

Sigma-Aldrich offers a convenient, ready-to-use kit that makes TVB determination faster and easier than ever. The kit contains all the reagents needed for 10 determinations of TVB according to the EU method. The suitability of each pre-made reagent was thoroughly tested and verified. Concentrations and specifications of the perchloric acid, sodium hydroxide and boric acid solutions were specially designed for this method. Using these pre-made solutions saves a great amount of time (**Table 1**) allowing the analyst to increase the number of samples analyzed per day. A fully detailed protocol is provided with the Kit.

Trimethylamine N-oxide (TMAO)

The second freshness indicator is trimethylamine N-oxide (TMAO). Seafood naturally contains TMAO, an osmolyte that is believed to counteract the deleterious effects of the high intracellular concentrations of urea in fish, sharks and rays. Ammonia and trimethylamine (TMA) are products of microbiological decomposition of TMAO. Some fish, even if stored on ice, also develop formaldehyde and dimethylamine (DMA) as decomposition products of TMAO.

Biogenic Amines Index

The biogenic amines index (BAI) is the third marker of seafood freshness. BAI is based on the fact that the amount of the biogenic amines histamine, putrescine and cadaverine increases steadily after the death of the fish due to bacterial action on amino acids.

The Official Food Control Authority of the Canton of Schaffhausen (Switzerland) has recently developed an ion chromatography method for the simultaneous detection and quantitative measurement of several amines, including DMA, TMA, TMAO, histamine, putrescine and cadaverine.^{1,2} The method is applicable to nitrogen concentrations from 5 mg/kg fish to at least 1500 mg/kg fish. Many different species of fish have been analyzed successfully, including plaice, redfish, codfish, halibut and flounder (analyses of the examined fish samples were performed by N.Seifert, Metrohm Ltd, Switzerland).

Table 1..... Components of TVB in Fish Kit and Kit componets

| Cat. No. | Brand | Product Name | Pack Size |
|----------|-------|--|-----------|
| 66662 | Fluka | TVB Fish Kit (enough for 10 analyses)* | 1 Kit |

Contents of TVB in Fish Kit (Cat. No. 66662)

| Cat. No. | Brand | Product Name | Concentration | Pack Size |
|----------|----------------|-----------------------------|-----------------------------|-----------|
| 08612 | Fluka | Perchloric acid solution | 6 g/100mL | 4 x 250mL |
| 55973 | Fluka | Sodium hydroxide solution | 20 g/100mL | 1 x 100mL |
| 55293 | Fluka | Boric acid solution | 3 g/100mL | 5 x 250mL |
| 85390 | Fluka | Silicone anti-foaming agent | 30g in 100mL water emulsion | 1 x 10mL |
| 34607 | Riedel-de Haën | Phenolphthaleine solution | 1 g/100mL 95 % ethanol | 1 x 30mL |
| 36083 | Riedel-de Haën | Tashiro Indicator | 3 g in 1L 95 % ethanol | 1 x 30mL |
| 09700 | Fluka | Ammonium chloride | > 99.5% | 1 x1g |

*Standard solutions are available in kit only and cannot be ordered individually.

(Biogenic amines continued)

The method is straightforward. Five grams of homogenized fish tissue and 50 mL buffer solution are combined and mixed. Each sample is prepared in duplicate. Calibration samples are prepared by spiking homogenized fish with 100, 300 or 500 µL of standard solution. An aliquot of the sample is transferred to an autosampler vial. Three different check standards are also prepared and one of the sample solutions is spiked with different concentrations of amines after the preparation to confirm the correct operation of the IC instrument. Seven-point calibration standards are prepared that bracket the expected concentrations in the samples. The solutions are stable for about one week in the refrigerator. The Kit contains all components for the analysis of TMAO and biogenic amines (Table 2).

The separation and measurement of the amines was performed on a 15cm Metrosep C2 IC column with 2cm guard, using nitric acid as eluent and indirect conductivity detection. The standards were injected twice. The sample solutions were injected five times each. The calculation was done by automatic integration using peak area for dimethylamine (DMA), trimethylamineoxide (TMAO), trimethylamine (TMA), putrescine, cadaverine and histamine. An example of the analysis of a standard solution of amines using this method appears in Figure 1.

A fully detailed protocol with chromatograms are provided with each kit showing amine standards and fresh and frozen plaice extracts.

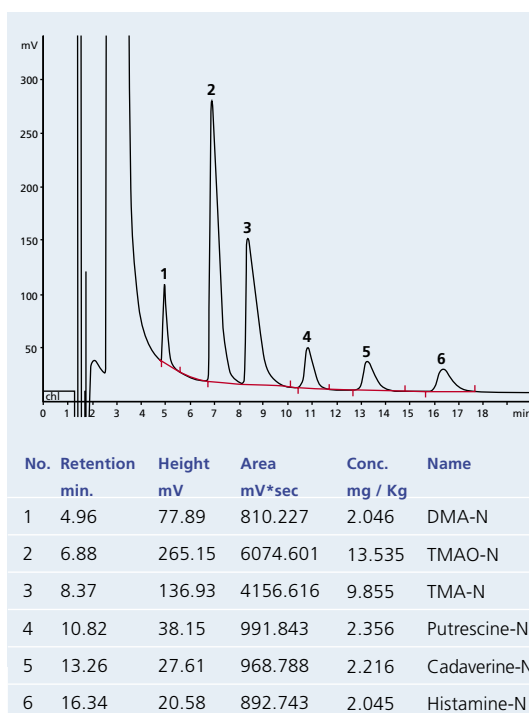


Figure 1..... Standard solution of amines (conditions were the same as the ones described as R. Oechsli, et al. J Chrom., submitted)

References

- [1] Diplomarbeit
- [2] G. Steil, Bestimmung von Qualitätsparameter von Dorschfilet (2002)
- [3] R. Oechsli, et al. J Chrom. (submitted)

Table 2..... TMAO and Biogenic Amines Kit and Kit Components

| Cat. No. | Brand | Product Name | Pack Size |
|----------|-------|------------------------------|-----------|
| 53851 | Fluka | TMAO and Biogenic Amines Kit | 1 Kit |

Contents of TMAO and Biogenic Amines Kit (Cat. No. 53851):

| | | | |
|-------|-------------|---|---------------------------------|
| 38270 | Riedel-Haën | Nitric acid standard solution concentrate | Enough for 1 L of 0.1N solution |
| 71180 | Riedel-Haën | Sodium acetate | 50g |
| 38960 | Fluka | Dimethylamine hydrochloride | 2g |
| 92277 | Fluka | Trimethylamine N-oxide dehydrate | 2g |
| 92270 | Fluka | Trimethylamine hydrochloride | 2g |
| 32810 | Fluka | Putrescine dihydrochloride | 2g |
| 53300 | Fluka | Histamine dihydrochloride | 2g |
| | Fluka | IC-column, C2 guard* | IC-column, C2 guard* |

*This column is available in kit only and cannot be ordered individually

Acknowledgment..... Fluka's R&D Department would like to thank to Laboratories of the Official Food Control Authority of the Canton of Schaffhausen for their support during the development of these Kits

New standards for Ion Chromatography.... Organic acids, ammonium and nitrite.

By Frederik Pillong, PhD, Product Manager, Fluka/Riedel-de Haën.....fpillong@sial.com

Fluka offers two different levels of quality documentation for our Ion Chromatography (IC) standards:

- **Standard Solutions from Fluka:** Produced of highest purity starting materials, filtered through 0.45µm membrane and Quality Assured to be accurate in concentration, this group is ideal for use as working standards. Some are traceable to the metrological Institutes EMPA/BAM or NIST.

- **Certified Reference Material:** These secondary reference materials are certified by two of 2 highly regarded metrological institutes: EMPA (Swiss Federal Laboratories for Materials Testing and Research) and BAM (German Federal Institute for Materials Research and Testing). A certificate of analysis is delivered with information about composition as measured by 2 or 3 different analytical methods, uncertainty of concentration and level of trace impurities. Each standard solution is sealed in a Mylar® bag to protect against evaporation and photodegradation.

New Organic Acids Standard Solutions for IC

Due to the omnipresence of organic acids in foods and beverages such as fruit drinks and wines and their link to certain nutritional, quality and safety aspects of the food, interest in measuring their concentration is growing rapidly. Ion chromatography is a common method for the analysis of organic acids.

To meet the requirements of our analytical customers, we developed a group of organic acids standards for ion chromatography (**Table 1**). The concentration is 1.000 g/L. All standards are carefully prepared from high purity starting materials, dissolved in water for analytical usage, filtered through a 0.22µm membrane filter to prevent column fouling and clogging. All standards are packaged in high density polyethylene bottles under sterile conditions. An expiration date is clearly provided on each bottle.

Table 1 New Organic Acids Standard Solutions for IC

| | Cat. No | Brand | IC standard | Quality | Concentration, pack size |
|---|---------|-------|-------------|-----------------------|--|
| ★ | 04924 | Fluka | Phthalate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 13669 | Fluka | Acetate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 38730 | Fluka | Citrate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 49897 | Fluka | Formate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 04621 | Fluka | Oxalate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 02717 | Fluka | Propionate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 50824 | Fluka | Tartrate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ | 49323 | Fluka | Malonate | Ready-to-use solution | 1.000g/L in water, 100mL polyethylene bottle |

★ New Product

Certified Reference Material Standards

For analysts who require the highest levels of documentation, certification and reliability, Fluka offers an extensive list of certified reference materials for ion chromatography (**Table 2**). Each anion is derived from the corresponding sodium salt and dissolved in high purity, 18MΩ, 0.22µm filtered water.

Table 2 Certified Anion Reference Standard Solutions for IC

| | Cat. No | Brand | IC standard | Salt | Quality | Concentration, pack size |
|--|---------|-------|-------------|----------------------------------|--|--|
| | 87603 | Fluka | Chloride | NaCl | Certified Reference Material for ion chromatography, certified by EMPA, according to ISO Guide 35 | 1.000g/L in water, 100mL polyethylene bottle |
| | 80218 | Fluka | Sulfate | Na ₂ SO ₄ | Certified Reference Solution, certified by EMPA, according to ISO Guide 35 | 1.000g/L in water, 100mL polyethylene bottle |
| | 87969 | Fluka | Bromide | NaBr | Certified Reference Material for ion chromatography, certified by EMPA, according to ISO Guide 35 | 1.000g/L in water, 100mL polyethylene bottle |
| | 86576 | Fluka | Nitrate | NaNO ₃ | Certified reference material for ion chromatography, certified by EMPA and BAM according to ISO Guide 35 | 1.000g/L in water, 100mL polyethylene bottle |
| | 80373 | Fluka | Fluoride | NaF | Certified Reference Material for ion chromatography, certified by EMPA, according to ISO Guide 35 | 1.000g/L in water, 100mL polyethylene bottle |
| | 81193 | Fluka | Phosphate | NaH ₂ PO ₄ | Certified Reference Material for ion chromatography, certified by EMPA, according to ISO Guide 35 | 1.000g/L in water, 100mL polyethylene bottle |

New Ionic Compounds Standard Solutions for IC

In addition to our existing anionic standards for ion chromatography, Fluka now offers two more inorganic standards: ammonium and nitrite (**Table 3**). These standards undergo the same manufacturing and QC procedures as the organic acids standards

Table 3 New Ionic Compounds Standard Solutions for IC

| Cat. No | Brand | IC standard | Salt | Quality | Concentration, pack size |
|---------|-------|-------------|---------------------------------|--|--|
| 17355 | Fluka | Bromide | NaBr | Ready to use solution, Traceable to EMPA/BAM | 1.000g/L in water, 100mL polyethylene bottle |
| 24889 | Fluka | Chloride | NaCl | Ready to use solution, Traceable to EMPA/BAM | 1.000g/L in water, 100mL polyethylene bottle |
| 27072 | Fluka | Chromate | K ₂ CrO ₄ | Ready to use solution, Traceable to EMPA/BAM | 1.000g/L in water, 100mL polyethylene bottle |
| 28491 | Fluka | Cyanide | Zn(CN) ₂ KCN | Ready to use solution, Traceable to EMPA/BAM | 1.000g/L in water, 100mL polyethylene bottle |
| 47070 | Fluka | Fluoride | NaF | Ready to use solution | 1.000g/L in water, 100mL polyethylene bottle |
| 72547 | Fluka | Nitrate | NaNO ₃ | Ready to use solution | 1.000g/L in water, 100mL polyethylene bottle |
| 79409 | Fluka | Phosphate | KH ₂ PO ₄ | Ready to use solution, Traceable to EMPA/BAM | 1.000g/L in water, 100mL polyethylene bottle |
| 86126 | Fluka | Sulfate | NaSO ₄ | Ready to use solution | 1.000g/L in water, 100mL polyethylene bottle |
| ★ 77012 | Fluka | Nitrite | NaNO ₂ | Ready to use solution, Traceable to NIST | 1.000g/L in water, 100mL polyethylene bottle |
| ★ 95023 | Fluka | Ammonium | NH ₄ Cl | Ready to use solution, Traceable to EMPA/BAM | 1.000g/L in water, 100mL polyethylene bottle |

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SPECTRANAL® High purity solvents and reagents for spectroscopy.

By Frederik Pillong, PhD, Product Manager, Sigma-Aldrich/Fluka.....fpillong@sial.com

We developed the SPECTRANAL® product line to meet the stringent purity requirements of modern UV, IR and fluorescence spectroscopy. SPECTRANAL® solvents are characterized by high UV transmittance, the absence of foreign band absorption in the IR range and very low content of endogenous fluorescent compounds. SPECTRANAL® carbon disulfide, carbon tetrachloride and 1,1,2-trichlorotrifluoroethane solvents are free of protons, having undergone tests to ensure the absence of compounds that might interfere with signals in ¹H-NMR spectroscopy.

For consistent product quality, we start with only the highest quality raw materials and take them through a stringent purification process using high efficiency distillation and non-distillation methods. Using high purity SPECTRANAL® solvents guarantees minimal interferences and no misleading spectral results.

Besides solvents the SPECTRANAL® line comprises other common spectroscopy chemicals. Potassium bromide (KBr) is mixed with the analyte substance to make the pellets needed for classical IR spectroscopy. Trifluoroacetic acid (TFA) is a common mobile phase additive for separation of synthetic peptides and peptide maps using HPLC-UV or HPLC-MS. SPECTRANAL® KBr and TFA are designed specifically for these applications.

The table below contains a selection of SPECTRANAL® solvents and reagents. For the complete list, please call us or visit our website:

www.sigma-aldrich.com/spectranal

Table 3 Selection of SPECTRANAL® Solvents and Reagents

| Cat. No | Brand | Compound | Specifications | Package Size |
|---------|----------------|--------------------------------|---|--------------------------------|
| 34932 | Riedel-de Häen | 1,1,2-Trichlorotrifluoroethane | >99.9% by GC | 2.5L, 4x2.5L |
| 34903 | Riedel-de Häen | N,N-Dimethylformamide | >99.9% by GC | 1L, 6x1L, 2.5L, 4x2.5L |
| 34905 | Riedel-de Häen | Carbon tetrachloride | >99.9% by GC | 1L, 6x1L, 2.5L, 4x2.5L |
| 34902 | Riedel-de Häen | Trichloromethane | Reag. ACS grade (>99%) | 1L, 6x1L, 2.5L, 4x2.5L |
| 34918 | Riedel-de Häen | 2,2,4-Trimethylpentane | >99.5% by GC | 2.5L, 4x2.5L |
| 34919 | Riedel-de Häen | Potassium bromide (KBr) | Passes test for suitability for IR spectroscopy | 100g, 6x100g |
| 34913 | Riedel-de Häen | N-Hexane | >97.0% by GC | 1L, 6x1L, 2.5L, 4x2.5L |
| 34957 | Riedel-de Häen | Trifluoroacetic acid | 99.8 % alkalimetric | 100mL, 6x100mL, 500mL, 6x500mL |
| 34936 | Riedel-de Häen | N-Heptane | > 99 % by GC | 1L, 6x1L, 2.5L, 4x2.5L |
| 34907 | Riedel-de Häen | Ethylene glycol | >99.8% by GC | 250mL, 1L, 2.5L |
| 34906 | Riedel-de Häen | Methanol | >99.9% by GC | 2.5L, 4x2.5L |



Special Offer!

Order any 2.5L bottle or multipack (4x2.5L, 6x1L, 6x100mL or 6x500mL), and get this special design cuvette rack (Cat. No. R2633)
Please quote Promotion Code 999 when placing your order.

Offer valid until 30th June 2005. Only 1 unit per customer.

What is the Difference Between *purum*, *puriss.* and *p.a.*? Fluka Purity Grades.

By Gert van Look, PhD, Head of Analytical Department of
Fluka Chemie (Buchs SG, Switzerland)
gvanlook@europe.sial.com

Product Specifications

Fluka and Riedel-de Haën operate with an integrated, process-oriented quality management system based on ISO 9001-2000. Clear product specifications are a central feature of our quality assurance procedure. Compliance with these specifications is carefully monitored by our quality control department to ensure that uniform high quality standards are met.

Explicit Specifications

All the data explicitly given on the subject of the appearance, content, activity, purity, impurities, suitability for application, special standards (e.g. ACS) and physical values are regarded as specifications.

Implicit Specifications

Certain qualities contain various implicit specifications. These always apply if no other details are given in the explicit specifications. Relevant details are found in **Table 1** (Definition of Purity Grades).

Purity Grades - General Classification

Purity grades

Fluka and Riedel-de Haën products are classified in a system of three purity grades:

- puriss.
- purum
- techn.

These grades are often supplemented with application-specific designations, such as "p.a.," standard, etc. or official designations such as ACS or DIN.

Further indications: *puriss.*, *p.a.*

The term "p.a." stands for *pro analysi*. The p.a. mention in the label denotes a product with guaranteed trace impurities and/or suitability for the indicated analytical application.

Table 1 Definition of Purity Grades

| Purity | Assay ^{a,b} | Ash ^c | Physical data | Appearance |
|---------|--------------------------------|------------------|---|---|
| puriss. | ≥ 98.5 % ^e | ≤ 0.1 % | according to catalog, specifications n ± 0.001, d ± 0.001 ^d | no extraneous color, homogenous appearance |
| purum | ≥ 95.0 % | ≤ 0.5 % | according to catalog, specifications n ± 0.001, d ± 0.002 ^d | slight extraneous color possible |
| techn. | Variable, but mostly ≥ 90 % | | according to catalog, specifications n ± 0.005, d ± 0.010 ^d | variations in color and shape possible |

a) Expressed in terms of dry substance

b) For optically active substances the assay refers to the sum of both enantiomers

c) Inorganic products and compounds with ash-forming elements are excluded

d) Where indications are given to two decimal points, ± 0.01 applies

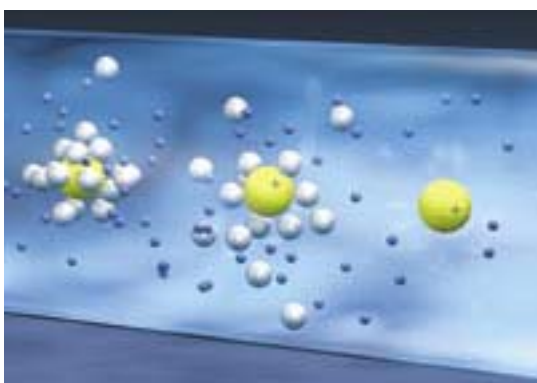
e) "*puriss. plus*" denotes an assay of ≥ 99.5 %

Table 2 Selection of *puriss.* and *puriss. p.a.* products

| Cat. No. | Brand | Product Name | Purity | Package Size |
|----------|-------|---------------------------------------|--|-------------------|
| 06220 | Fluka | Aluminum chloride | puriss., p.a., anhydrous, crystallized, ≥ 99.0 % AT | 100g, 1 kg, 2.5kg |
| 30631 | Fluka | Sodium 1-decanesulfonate | puriss., p.a., for ion pair chromatography, ≥ 99.0 % Titration | 2.5g, 10g, 50g |
| 40248 | Fluka | N,N-Dimethylformamide | puriss., absolute, over molecular sieve (H ₂ O ≤ 0.005%) ≥ 99.8 % GC | 250mL, 1L, 2.5L |
| 52765 | Fluka | Hexane | puriss., p.a., ACS, ≥ 99.0 % GC | 1L, 2.5L, 25L |
| 52862 | Fluka | Sodium 1-hexanesulfonate monohydrate | puriss., p.a., for ion pair chromatography, ≥ 99.0 % Titration | 2.5g, 10g, 50g |
| 56485 | Fluka | N-Hydroxysulfosuccinimide sodium salt | puriss., ≥ 98.5 % HPLC | 250mg, 1g, 5g |
| 59300 | Fluka | 2-Propanol | puriss., p.a., ACS, ≥ 99.8 % GC | 1L, 2.5L, 25L |
| 64340 | Fluka | DL-Methionine | puriss., ≥ 99.0 % NT | 100g, 500g, 1kg |
| 65543 | Fluka | Methanol | puriss., p.a., absolute, ACS, ≥ 99.8 % GC | 1L, 2.5L, 5L, 25L |
| 87371 | Fluka | Tetrahydrofuran | puriss., absolute, over molecular sieve (H ₂ O ≤ 0.005%), 99.5% GC | 250mL, 1L, 2.5L |
| 91707 | Fluka | Trifluoroacetic acid | puriss., p.a., for HPLC ≥ 99.0 % GC | 10 1mL ampoules |

Suitability of Common Solvent Grades for LC-MS Analysis Benefits of LC-MS CHROMASOLV® Solvents.

By Nour Kayali, PhD, Mass Spectrometry Service, Chemistry Faculty, Complutense University, Madrid, Spain
kayali@quim.ucm.es



Because of its sensitivity and specificity, LC-MS is one of the fastest growing segments in analytical sciences. However, impurities in mobile phase solvents can lead to high background and seriously diminish the ability to detect and identify compounds that exist at very low levels. Furthermore, particulate and non-volatile impurities can clog susceptible and delicate hardware components, necessitate time-consuming recalibration and cause instrument down-time. Special LC-MS grade solvents are necessary to meet the requirements of high sensitivity LC-MS instruments and analyses.

Acetonitrile is the most commonly used LC-MS and LC-UV mobile phase solvent. Solvent suppliers offer many grades of acetonitrile, each delivering a minimum/maximum baseline rise under certain detection and gradient or isocratic flow profiles. Comparison of the spectra provided by different grades of acetonitrile can be seen in figures a, b and c. All spectra were obtained using direct introduction into a Bruker Daltonics Esquire-LC ESI-Ion Trap MSn System. Even the solvent with the most stringent UV grade specifications, SupraGradient grade (Figure 1a, maximum transparency to 190 nm), gave spectra with too high background to allow MS detection.

However, the LC-MS grade solvent, Riedel de Haën's LC-MS CHROMASOLV®, provides acceptably low UV and MS backgrounds (Figure 1c). Levels of some interfering ions were lower than competitive solvent (Figure 1b). The low levels of alkali impurities in LC-MS CHROMASOLV® also reduce cluster ion formation.

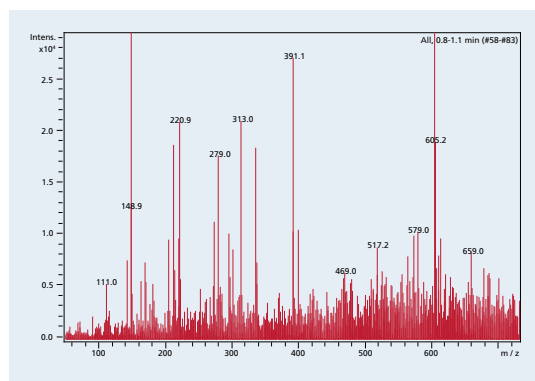


Figure 1a SupraGradient acetonitrile

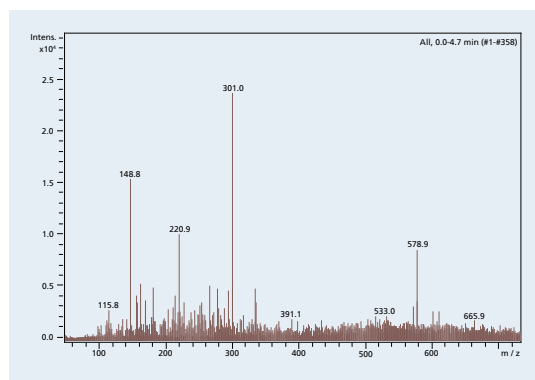


Figure 1b Competitive LC-MS acetonitrile

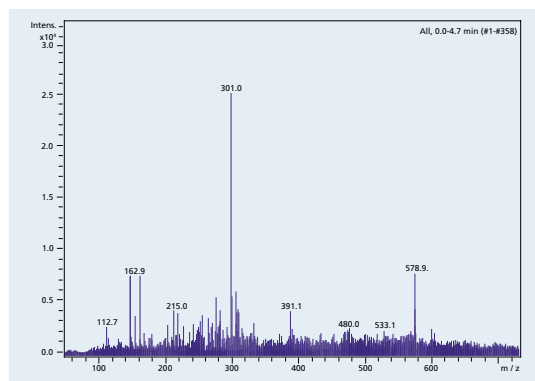


Figure 1c LC-MS CHROMASOLV® acetonitrile (Cat. No. 34967)

Figure 1 Comparison of MS spectra obtained using by different grades of acetonitrile

Another important feature of the LC-MS CHROMASOLV® solvents is the significantly reduced levels of the omnipresent contaminant, phthalate. Phthalate is found in many solvents by leaching from storage containers or by improper handling. To ensure the LC-MS solvent does not become contaminated by phthalates during the transfer process, it is recommended to use Sigma-Aldrich's PTFE Dispensing Aid Adapter (Cat. No. 79212).

Test Products: LC/MS CHROMASOLV® Products for You to Test Drive

LC-MS CHROMASOLV® line of solvents and blends is competitively priced and tested for suitability *in situ*. These high purity solvents are an exceptional value and belong in every serious analytical laboratory. Find out if the LC-MS CHROMASOLV® meets your most demanding needs of LC-MS solvents by taking advantage of our free, no obligation evaluation offer. Order any solvent from the list below, quoting the Promotion Code 997.

Offer limited to only one unit per customer. Offer valid until June 30, 2005.

Table 1: Pure Solvents

| Cat. No. | Brand | Pure Solvent | Pack size | Packaging |
|----------|----------------|---------------------------------|-----------------------|--------------------|
| 39253 | Riedel-de Häen | Water LC-MS CHROMASOLV® | 1L | white glass bottle |
| 34967 | Riedel-de Häen | Acetonitrile LC-MS CHROMASOLV® | 1L, 6X1L, 2.5L 4X2.5L | amber bottle |
| 34966 | Riedel-de Häen | Methanol LC-MS CHROMASOLV® | 1L, 6X1L, 2.5L 4X2.5L | amber bottle |
| 34965 | Riedel-de Häen | 2-Propanol LC-MS CHROMASOLV® | 1L, 6X1L, 2.5L 4X2.5L | amber bottle |
| 34972 | Riedel-de Häen | Ethyl acetate LC-MS CHROMASOLV® | 1L, 2.5L | amber bottle |

Table 2: Solvent Blends

| Cat. No. | Brand | Solvent Blend | Pack size | Packaging |
|----------|----------------|---|-----------|--------------|
| 34978 | Riedel-de Häen | Water with 0.1% TFA LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34976 | Riedel-de Häen | Acetonitrile with 0.1% TFA LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34974 | Riedel-de Häen | Methanol with 0.1% TFA LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34673 | Riedel-de Häen | Water with 0.1% formic acid LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34668 | Riedel-de Häen | Acetonitrile with 0.1% formic acid LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34671 | Riedel-de Häen | Methanol with 0.1% formic acid LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34675 | Riedel-de Häen | Water with 0.1% acetic acid LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34678 | Riedel-de Häen | Acetonitrile with 0.1% acetic acid LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34672 | Riedel-de Häen | Methanol with 0.1% acetic acid LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34674 | Riedel-de Häen | Water with 0.1% ammonium acetate LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34669 | Riedel-de Häen | Acetonitrile with 0.1% ammonium acetate LC-MS CHROMASOLV® | 2.5L | amber bottle |
| 34670 | Riedel-de Häen | Methanol with 0.1% ammonium acetate LC-MS CHROMASOLV® | 2.5L | amber bottle |

Table 3: Teflon Adapter

| Cat. No. | Brand | Description | Packaging |
|----------|----------------|----------------------|-----------|
| 79212 | Riedel-de Häen | Dispensing Aid Agent | 1 unit |



Picture

LC-MS bottles PTFE adaptor (Cat. No 79212)

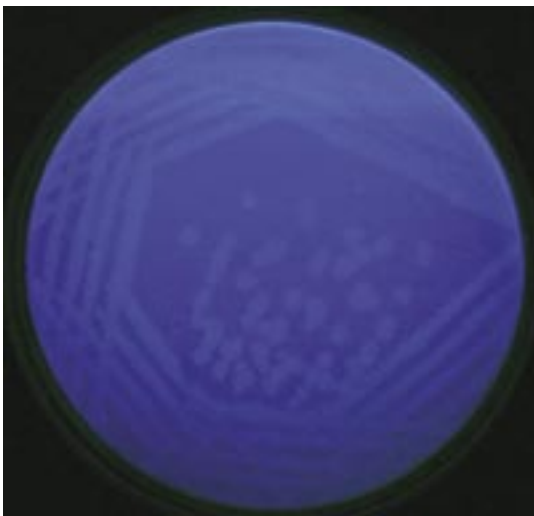
HiFluoro™ Pseudomonas Agar Base HiFluoro™ Pseudomonas Agar Base is used as a selective medium for the isolation of *Pseudomonas aeruginosa* from diverse samples. The presence of these bacteria is readily visualized by simply observing the culture under long wave UV light.

By Jvo Siegrist, Product Manager, Fluka/Riedel-de Haën isiegris@sial.com

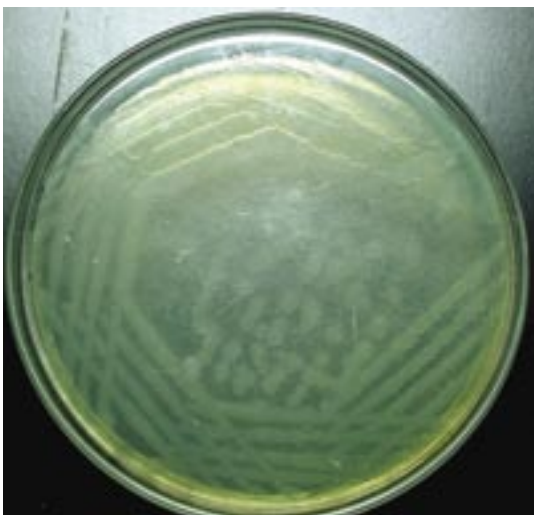
HiFluoro™ Pseudomonas Agar Base is a selective media that includes specific fluorogenic substrate. It is based on the formula described by King et al. (1). *Pseudomonas aeruginosa* cleaves the fluorogenic compound in the agar to release fluorogen which produces a visible fluorescence under long wave UV light (Pictures 1 and 2).

Reference

- [1] E.O. King, et al., "Media for the demonstration of phycocyanin fluorescein," J. Lab. Clin. Med. 44, 301 (1954)



Picture 1 *Pseudomonas aeruginosa* on HiFluoro™ Pseudomonas Agar under UV light



Picture 2 *Pseudomonas aeruginosa* on HiFluoro™ Pseudomonas Agar

Table 1.... Ordering Information

| Cat. No. | Brand | Description | Pack size |
|----------|-------|---------------------------------|------------|
| 78996 | Fluka | HiFluoro™ Pseudomonas Agar Base | 100g, 500g |

Microbiology Reagents Sigma-Aldrich offers a diverse line of biochemical reagents that are sensitive and selective for the metabolic fingerprints microbiologists use to identify species of bacteria.

By Jvo Siegrist, Product Manager, Fluka/Riedel-de Haën jsiegris@sial.com

Although there are many high tech instruments used in today's microbiological laboratories, scientists still rely on the classical biochemical tests where a simple color change identifies the presence of certain microorganisms. Most of these reactions are based on an enzyme-substrate reaction. There are also methods to detect certain microorganisms by their metabolites

using chemical reactions and/or complexation techniques. The reagents give a color change that is used as evidence to help identify an unknown microorganism or confirm the presence of a known one. Sigma-Aldrich offers many of these detection reagents, the most popular of which are presented in **Table 1** below.

Table 1 Biochemical Tests

| Cat. No. | Brand | Reagent | Detection of | Differentiation of Organisms | Reaction | Color (positive result) |
|----------|-------|--|---------------------------------|---|--|-------------------------|
| 29333 | Fluka | Barritt's Reagent A | Acetoin production | <i>Klebsiella</i> , <i>Enterobacter</i> | Oxidize acetoin, reacts with alpha-naphthol | Red |
| 39442 | Fluka | Barritt's Reagent B | Acetoin production | <i>Klebsiella</i> , <i>Enterobacter</i> | Oxidize acetoin, reacts with alpha-naphthol | Red |
| 07689 | Fluka | O'Meara's Reagent | Acetoin production | <i>Klebsiella</i> , <i>Enterobacter</i> | Oxidize acetoin, reacts with alpha-naphthol | Red |
| 38497 | Fluka | Nitrate Reagent A | Reduction of nitrate to nitrite | <i>Enterobacter</i> , <i>E. coli</i> , <i>Salmonella</i> | Reacts with sulphanilic acid, forms an azo dye with alpha-naphthylamine | Red |
| 39441 | Fluka | Nitrate Reagent B | Reduction of nitrate to nitrite | <i>Enterobacter</i> , <i>E. coli</i> , <i>Salmonella</i> | Reacts with sulphanilic acid, forms an azo dye with alpha-naphthylamine | Red |
| 07345 | Fluka | Oxidase Reagent acc. Gaby-Hadley A | Oxidase activity | Oxidase positives and negatives | Oxidize dimethyl-p-phenylenediamine hydrochloride, build a complex with alpha-naphthol | Dark blue |
| 07817 | Fluka | Oxidase Reagent acc. Gaby-Hadley B | Oxidase activity | Oxidase positives and negatives | Oxidize dimethyl-p-phenylenediamine hydrochloride, build a complex with alpha-naphthol | Dark blue |
| 18502 | Fluka | Oxidase Reagent acc. Gordon-McLeod | Oxidase activity | Oxidase positives and negatives | Oxidize N,N-dimethyl-p-phenylenediamine to Wurster's blue | Dark blue-purple |
| 08714 | Fluka | Methyl Red Solution | Strong acid production | <i>E. coli</i> , <i>Enterobacter</i> | pH indicator reaction | Red |
| 80353 | Fluka | TDA Reagent | Tryptophan deaminase activity | <i>Proteus</i> | Reacts with indolepyruvic acid | Dark brown |
| 49825 | Fluka | DMACA Reagent | Tryptophanase activity | <i>E. coli</i> | Reacts with indole | Blue purple |
| 60983 | Fluka | Kovac's Reagent for indoles | Tryptophanase activity | <i>E. coli</i> | Reacts with indole | Red |
| 67309 | Fluka | Kovac's Reagent for indoles (improved formulation) | Tryptophanase activity | <i>E. coli</i> | Reacts with indole | Red |

Application example: Oxidase Reagents according to Gaby-Hadley

Gaby-Hadley reagents are used to perform the cytochrome-c oxidase test via different methods. Verifying the presence or absence of cytochrome-c oxidase activity is one of the first and most important steps in bacterial identification process. The cytochrome-c oxidase present in most gram negative bacteria oxidizes dimethyl-p-phenylenediamine and alpha-naphthol to a dark blue complex (indophenol blue). The test can be used in agars in broths, plates and on discs (see Figure 1-4). It is good practice to run both positive and negative controls to confirm the results.

Instructions:

Grow test culture on nutrient agar slant for 18-24 hours. Add 2-3 loops of reagents A and B to slant. Tilt tube to permit reagents to mix and flow over growth.

Result:

Color change occurs within 2 minutes. A positive reaction shows a dark purple blue color on the growth, especially at the border of the colony.

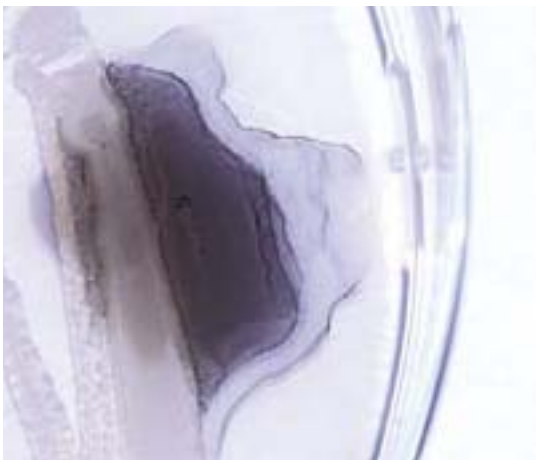


Figure 1 *E.coli* on a agar plate

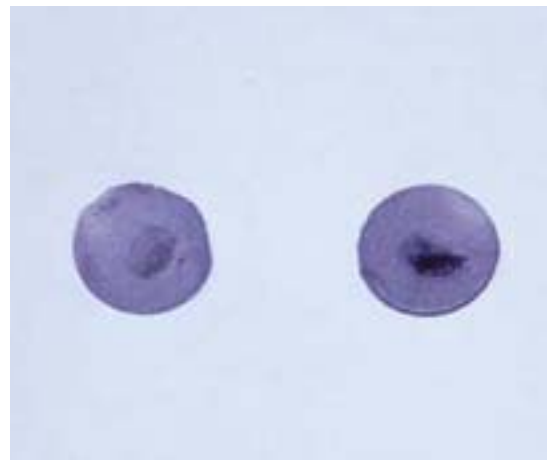


Figure 3 Paper discs impregnated with 2 drops of each reagent. Left disc with *E.coli*. Right disc with *Pseudomonas fluorescens*.

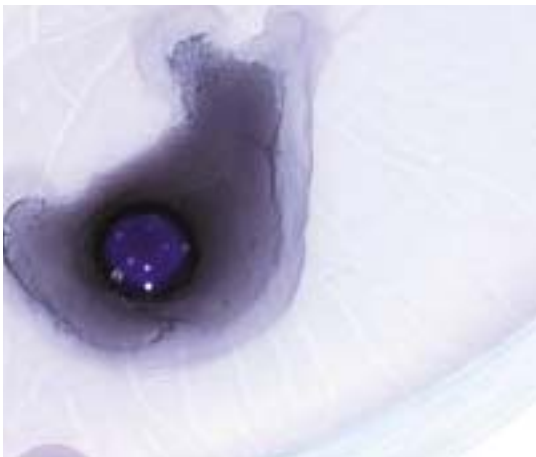


Figure 2 *Pseudomonas fluorescens* on a agar plate

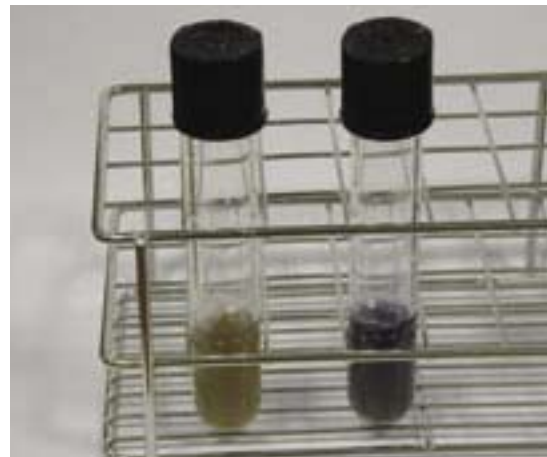


Figure 4 Cultures with 2 drops of each reagent. Left tube with *E.coli*. Right tube with *Pseudomonas fluorescens*.

New Microbiology CD Microbiology Media Data Bank CD is a valuable tool for all microbiologists.

This CD contains a complete listing of growth media, base ingredients, supplements, reagents, discs, strips and tests for microbiology from Fluka and Sigma brands. An improved and advanced search tool allows you to search for names, synonyms, keywords and the product numbers of Fluka, Sigma and other suppliers as well, making it an excellent tool for all microbiologists. You will find a complete Microbiology Media Data Bank on this CD which gives detailed product information, including:

- Product composition
- Directions for use
- Required additives
- Test strains
- Package sizes
- Background information about the application
- References and literature

The detailed product information is printable as a working sheet. A link to our website gives you the prices in your local currency. But you must hurry; the stock of this valuable CD is limited. Please reserve your CD now by registering on-line or returning the enclosed reply card (Literature Code: HIG) or go to our website: www.sigma-aldrich.com/microbiology_cd



New! HYDRANAL® Multimedia Guide Version 2.0 Get your free copy by completing the attached form or visiting our website

Moisture determination is a ubiquitous analytical need, and Karl Fischer titration is one of the most commonly-employed techniques. Sigma-Aldrich introduced the HYDRANAL® product line over 25 years ago and since then it has become the industry standard for quality, capacity, speed, safety and reliability in moisture determination. Now you can learn about HYDRANAL® and the theory behind it with our new HYDRANAL® Multimedia Guide. In it you'll find a description of the technology, product overview and a database with over 500 moisture analysis applications.

The HYDRANAL® Multimedia Guide contains:

- Theory behind volumetric and coulometric Karl Fischer titration
- Overview of the HYDRANAL® product line and a complete product listing
- Introduction on how to prepare and analyze samples for moisture analysis using HYDRANAL®
- Database of more than 500 HYDRANAL® applications
- Bibliography of HYDRANAL® citations in the scientific literature
- How to request Laboratory Reports, MSDS and other literature, and get technical help from our expert HYDRANAL®-staff



Multimedia Guide version 2.0 is currently available in German, English and Spanish.

To obtain your copy of the HYDRANAL® Multimedia Guide, please complete the Reply Card in this issue of Analytix, or go to our website: (Literature Code: GVJ) www.sigma-aldrich.com/hydranal_cd

HYDRANAL®-Moisture Testkit..... Simple, rapid, portable and economical moisture determination without expensive equipment.

By Michael Jeitziner, Product Manager, Fluka/Riedel-de Haën mjeitziner@sial.com

When you want a quick, portable method to measure a sample's moisture content, for example in field sampling operations, the **HYDRANAL®-Moisture Testkit** may be just what you need. The Testkit works on the Karl Fischer principle, like the rest of our **HYDRANAL®** line. Basically, sulfur dioxide is oxidized in the presence of iodine with ensuing consumption of water; excess iodine gives the characteristic yellow color. The end point of the titration is indicated by a sharp color change. Water levels from 0.001% to (>10%) in samples from 10µL to 20mL can be analyzed using this Testkit. Depending on the amount of water present, it is possible to carry out double or triple measurements in a single titration vessel.

The operation is simple (**Figure 1**). Pre-weighed sample is added to the provided glass vessel. **HYDRANAL®-Solvent E** is added and the vessel is sealed tightly and shaken. With the provided syringe, **HYDRANAL®-**

Titrant Reagent (Cat. No. 37857) is slowly added through the septum with shaking. When the abrupt color change to yellow occurs, all of the water in the sample has been titrated signaling the endpoint. By measuring the volume of **HYDRANAL®-Titrant Reagent** added, and knowing the mass or volume of sample and the titre of the titration solution, the % moisture can be easily determined.

The test kit uses safe, non-toxic and easily disposed of ethanol-based reagents. Each box contains two 500 mL bottles of **HYDRANAL®-Solvent E**, **HYDRANAL®-Titrant 5E** in a 100 mL glass bottle with septum, and one 100 mL bottle **HYDRANAL®-Standard 5.00** for titre determination. The kit also contains a funnel for pouring the Solvent (Cat. No. 34730) into the bottle which acts as the titration vessel, and 1mL and 5mL syringes and needles.

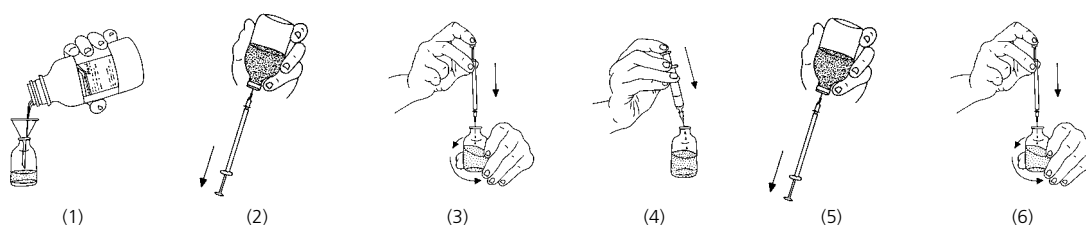


Figure 1 Simple, manual operation of **HYDRANAL®-Moisture Testkit**

- (1) Filling with **HYDRANAL®-Solvent E**
- (2) Filling of the titration syringe
- (3) Pre-titration
- (4) Application of the sample
- (5) Filling with **HYDRANAL® Titrant**
- (6) Titration

Table 1.... Ordering Information

| Cat. No. | Brand | Product | Use |
|----------|----------------|--|---|
| 37858 | Riedel-de Haën | HYDRANAL®-Moisture Testkit | Test kit for the visual determination of moisture content |
| 37857 | Riedel-de Haën | HYDRANAL®-Titrating Reagent for Testkit | refill pack for HYDRANAL®-Moisture Testkit |
| 34730 | Riedel-de Haën | HYDRANAL® Solvents E | 500mL |
| 34813 | Riedel-de Haën | HYDRANAL® Standards 5.00 | 100mL |

New HYDRANAL®-Medium K For reliable moisture determination in aldehydes and ketones without toxic components.

By Helga Hoffmann, Technical Support HYDRANAL® Manager..... hhoffman@europe.sial.com

HYDRANAL®-Medium K is a new product for the determination of water content in aldehydes and ketones using the Karl Fischer method. It has advantages over other reagents with respect to application, toxicity and transportation requirements.

If you have used HYDRANAL®-Working Medium K (Cat. No. 34817), the special medium for aldehydes and ketones, you will see how new HYDRANAL®-Medium K has a distinct safety advantage. Because HYDRANAL®-Working Medium K contains 2-chloroethanol, it is categorized as T+, Very Toxic. However, in new HYDRANAL®-Medium K we replaced the 2-chloroethanol with considerably less hazardous alcohols. Rather than toxic, it is categorized as X_n, Harmful, due to the presence of chloroform.

This improvement in safety does not adversely change the operation or performance of the new HYDRANAL®-Medium K. During development we verified that HYDRANAL®-Medium K is just as reactive as HYDRANAL®-Working Medium K and it guarantees the same short titration times. Using HYDRANAL®-Composite 5K as the titration reagent, we carried out comparison titrations in both media, titrating a large variety of different aldehydes and ketones, including acetone, 4-methyl-2-pentanone, octanone, cyclohexanone, acetophenone, 2-acetylpyridine, 2-pyrrolidinone, diacetyl, 1,3-cyclohexandione, pyruvic acid, levulinic acid, acetaldehyde, paraldehyde, 3-hydroxybenzaldehyde, anisaldehyde and others. The parameters we evaluated were:

- Capacity for the substance in 30mL of medium
- Repeatability of water content, mean value and standard deviation
- Duration of titration
- Accuracy of the recovery of added water after the titration of a sample

Another advantage of the new HYDRANAL®-Medium K formulation is seen with very reactive aldehydes, such as propionaldehyde, butyraldehyde and crotonaldehyde. Other compounds, such as salicylaldehyde, acetylacetone 2,4-dihydroxyacetophenone and 2-benzoylpyridine showed enhanced capacity and accuracy. The unique alcohols in the new HYDRANAL®-Medium K formulation also give it different solvation properties compared to the original formulation. An example of this difference is seen with the compound 3-benzoylpropionic acid, 5g of which can be dissolved easily in the new formulation.

Overall, from safety and performance standpoints the new HYDRANAL®-Medium K offers distinct advantages to the user making it the ideal choice for sensitive moisture determinations in aldehydes and ketones.

The complete listing of the HYDRANAL® product line can be found at:

www.sigma-aldrich.com/hydranal



Table 1 HYDRANAL® - Medium K product listing

| Cat. No | Brand | Product | Package size |
|---------|----------------|---------------------------|-----------------|
| 34698 | Riedel-de Haën | HYDRANAL® - Medium K | 1L |
| 34816 | Riedel-de Haën | HYDRANAL® - Composite 5 K | 500mL, 1L, 2.5L |

New Product Corner 5 Liter VOLPAC® containers of volumetric, ready-to-use solutions.

By Michael Jeitziner, Product Manager, Fluka/Riedel-de Haën mjeitziner@sial.com

If you routinely use large volumes of common buffers, solutions, reagents and dilute acids or bases, VOLPAC® containers will save preparation time, and guarantee consistency, purity and accuracy.

The 5 liter and 10 liter VOLPAC® containers consist of a flexible polyethylene bag with an outlet tap and a cubic cardboard supporting frame. As the volume is depleted the design of the bag prevents air entering the bag, eliminating the possibility of contamination.



Figure VOLPAC® Containers

Advantages of VOLPAC® containers include:

- **High quality of contents and packaging**
- **Easy handling**
- **No contamination during use**
- **Small space requirement**
- **Reduced packing material**
- **Easy disposal of empty containers**

Table 1 Selection of products in VOLPAC® containers

| Cat. No | Brand | Description | Package size |
|---------|----------------|---|--------------|
| 33643 | Riedel-de Haën | Buffer solution pH 4.0 (20 °C) (citric acid / sodium hydroxide solution / sodium chloride) solution ready for use with fungicide | 5L, 10L |
| 33646 | Riedel-de Haën | Buffer solution pH 7.0 (20 °C) (potassium dihydrogen phosphate / di-sodium hydrogen phosphate) solution ready for use with fungicide | 5L, 10L |
| 33648 | Riedel-de Haën | Buffer solution pH 9.0 (20 °C) (borax / hydrochloric acid) solution ready for use | 5L, 10L |
| 35375 | Riedel-de Haën | Silver nitrate solution, Ph Eur standard solution (volumetric) 0.1M | 5L, 10L |
| 34544 | Riedel-de Haën | IDRANAL® B, IDRANAL® III solution with zinc complex added, for water hardness determination (1 mL = 1 German degree of hardness in 100 mL of water) | 5L, 10L |
| 34550 | Riedel-de Haën | IDRANAL® III standard solution (EDTA Na ₂ solution) Ph Eur, for metal titration solution (volumetric) 0.1 M | 5L, 10L |
| 35102 | Riedel-de Haën | IDRANAL® III standard solution solution (volumetric, EDTA Na ₂ solution) 0.2M | 5L, 10L |
| 35103 | Riedel-de Haën | IDRANAL® IV standard solution (CDTA solution) Ph Eur, for metal titration solution (volumetric) 0.1M | 5L, 10L |
| 34631 | Riedel-de Haën | Potassium dichromate solution for COD determination according to DIN 38409, part 41 standard solution (volumetric) 0.02M | 5L, 10L |
| 35245 | Riedel-de Haën | Sodium thiosulfate solution, Ph Eur standard solution (volumetric) 0.1M | 5L, 10L |
| 35256 | Riedel-de Haën | Sodium hydroxide standard solution, Ph Eur solution (volumetric) 1.0M | 5L, 10L |
| 35257 | Riedel-de Haën | Sodium hydroxide standard solution, solution (volumetric) 0.5M | 5L, 10L |
| 35263 | Riedel-de Haën | Sodium hydroxide standard solution, Ph Eur solution (volumetric) 0.1M | 5L, 10L |
| 35328 | Riedel-de Haën | Hydrochloric acid standard solution, Ph Eur solution (volumetric) 1.0M | 5L, 10L |
| 35335 | Riedel-de Haën | Hydrochloric acid standard solution, Ph Eur solution (volumetric) 0.5M | 5L, 10L |
| 35335 | Riedel-de Haën | Hydrochloric acid standard solution, Ph Eur solution (volumetric) 0.1M | 5L, 10L |
| 35357 | Riedel-de Haën | Sulfuric acid standard solution, Ph Eur solution (volumetric) 0.1M | 5L, 10L |
| 35358 | Riedel-de Haën | Sulfuric acid standard solution Ph Eur solution (volumetric) 0.05M | 5L, 10L |

New Product Corner New reference electrode for ion-selective electrodes.

By Michael Jeitziner, Product Manager, Fluka/Riedel-de Haën mjeitziner@sial.com

This reference electrode is designed specifically for use with ion selective electrodes. It is reliable and accurate even when ion concentrations are very low. The electrode has two openings for filling and a exchanging the electrolyte solution. (Double-junction Ag/AgCl reference electrode, cable not included.)

Technical Specifications:

- Reference electrolyte: Refillable, 3M KCl
- Reference system: Ag/AgCl cartridge, Ag⁺-free
- Operating range: 0 - 80 °C (32 - 176 °F)
- Diaphragm: Teflon
- Shaft material: Glass
- Electrode plug: S7
- Application: Reference for ion-selective electrodes

Figure 1 ISE Reference Electrode



Table 1 Ordering information

| Cat. No | Brand | Product | Package size |
|---------|-------|---|--------------|
| 16811 | Fluka | Reference electrode for ion-selective electrode | 1 unit |
| 16613 | Fluka | Electrode cable with S7/BNC-Connector for reference electrode | 1 unit |
| 30676 | Fluka | Electrode cable with S7/DIN-Connector for reference electrode | 1 unit |
| 60137 | Fluka | Electrolyte filling solution (3M KCl for Ag/AgCl electrodes) | 250mL |
| 45137 | Fluka | Electrode body ISE | 1 unit |
| 44765 | Fluka | Tool for removing membranes | 1 unit |

New Product Corner Büchi-Fluka NIR Spectral Library

By Ing. Harald Agrinz, Chemist of Fluka Chemie GmbH (Buchs SG, Switzerland) hagrinz@europe.sial.com

Near Infrared Spectroscopy is a very powerful method for fast identification of substances; unfortunately, in many cases you have to build up a calibration or library before you can use it.

The Büchi-Fluka NIR Library is a collection of more than 4000 Near Infrared spectra of solids a representative cross section of the Fluka chemicals. You receive a single file for each spectrum, stored in jcamp-dx file format for easy import into your own spectroscopy software. Each spectrum includes the Fluka product number, chemical name and lot number of the measured substance. In addition, you receive a text file with Fluka product numbers and chemical names and a Microsoft® Excel file for the correlation of product number, name and CAS number.

Fluka production uses this library together with a Büchi NIRVIS® spectrometer at the receiving dock to identify and compare uniformity of incoming chemical substances automatically and within seconds. You can use it to search unknown substances, improve your own library or just visually compare your spectra with our library spectra.

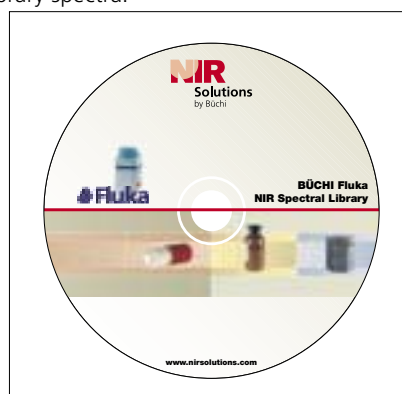


Table 1 Ordering information

| Cat. No | Brand | Product |
|---------|-------|--|
| 18378 | Fluka | NIR Spectra library Büchi-Fluka on CD |

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