Authenticity of 100% Orange Juice in the Czech Market in 1996–2001

MICHAL VOLDŘICH[1], PETRA SKÁLOVÁ[1], FRANTIŠEK KVASNIČKA[1], PETR CUHRA[2], MARTIN KUBÍK[2] and PETR PYŠ

[1]Department of Food Preservation and Meat Technology, Institute of Chemical Technology, Prague; [2]Czech Agricultural and Food Inspection, Prague, Czech Republic

Abstract

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Commercial orange juices samples obtained from the Czech market were analysed in the years 1996–2001. The quality and authenticity of samples was evaluated according to the Code of Practice of AIJN (selected main analytical criteria were followed – K^+, Na^+, Ca^{2+}, Mg^{2+}, citric, isocitric and malic acid, citric acid/isocitric acid ratio, glucose, fructose, saccharose, sorbitol, formol number, flavonoid glycosides – according to Davis and HPLC procedure, refractive index and other). The approach of producers to the quality and authenticity of juices developed during the years of observation. The main cases of adulteration in 1996 were as follows: (i) lower fruit content in juice, (ii) massive addition of sugars masked with addition of citric acid, (iii) several examples of "synthetic" orange juice were found. In the subsequent years the authenticity slightly improved, the main problems in 2000/2001 were: (i) lower refractive index, (ii) pulp wash addition, (iii) lower quality of water used for juice reconstitution, (iv) undeclared addition of sugar. The deviations were found not only in the case of the juices of Czech producers, but also in several discount and low-end products of foreign producers. Possible ways of improving the quality and authenticity are discussed (e.g. the preparation of a Czech National Standard taking over the requirements for juices and nectars according to the Code of Practice of the AIJN, Participation in the European Quality Control System [EQCS], etc.).

Keywords: orange juice; authenticity; adulteration; fruit content

Fruit juices and nectars represent a food commodity that is very often subjected to adulteration. The development of adulteration procedures corresponded with increase in the volumes of production. The first big cases of fruit juice and nectar adulteration were in the fifties in the USA, where the volume of production was higher than the amount of fruit available (NAGY *et al.* 1988). The adulteration of fruit juices is also a problem in Europe, but during the years the volume of adulterated juices and nectars decreases due to the activities of the national fruit juice industries and their federation. This activity has been prevented in the EU, where the Association of the Industry of Juices and Nectars from Fruits and Vegetables of the EEC (AIJN) was established. One of the principle activities of the AIJN was the development of reference guidelines that were prepared on the basis of already existing information (RSK and AFNOR standards), such as national standards as well as literature and experience. The guidelines The Code of Practice of the AIJN, the first version was issued and approved by the European industry in 1990 and now consists of 19 reference guidelines: orange, grapefruit, apple, grape, pineapple, lemon, passion fruit, pear, apricot, tomato, black currant, sour cherry, raspberry, strawberry, peach, mango, guava, banana and mandarin (GREEVE 1998). Each of the 19 reference guidelines of the Code of Practice contains general explanatory notes, basic (obligatory) quality requirements such as min. relative density and corresponding Brix values, isotopes, biogenic acids, ethanol, arsenic and heavy metals and the essential parameters and their characteristic value or range for the evaluation of identity and authenticity as well as some recommended quality criteria for juices, e.g. acids, minerals, sugars, amino acids, flavonoids, etc. (AIJN 1999).

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In the Czech Republic the situation with authenticity of fruit juices is similar as it was in Europe several years ago. Legal rules for juices and nectars (Decree No. 335/1997 as amended by Decree No. 45/2000, Act No. 110/1997 amended by Act No. 306/2000 and relevant decrees) provide vague definitions of juices, permitted treatments, permitted ingredients and additives, contaminant levels, labelling requirements and there is no definition of the authenticity and identity of a juice. Recently Czech national standards based on the AIJN guidelines for orange, apple and grapefruit juices have been prepared (ČSN 5685 41 – Fruit and vegetable juices – orange juice, ČSN 5685 42 – Fruit and vegetable juices – grapefruit juice and ČSN 5685 43 – Fruit and vegetable juices – apple juice), the standards have no effectuality but could be used in the legal action. Other factors affecting the authenticity and quality of fruit juices are: continuing economic recession, not very clear competition in the market of juices, pressure of distribution chains, import of juices of lower quality (some EU producers often produce "special" products for the Czech market not complying with AIJN), ongoing development of the market.

In the Czech market there is a relatively high number of adulterated or non-authentic fruit juices declared as 100% fruit juices. The main deviations are: lowering of fruit content (addition of sugars, acids, artificial mixtures), unlabelled sugar addition (usually without lowering of fruit content), pulp wash addition (including pure pulp wash juices), lower refractive index, low quality of water used for reconstitution and others.

Since 1996 the Association of Czech Producers of Fruit Juices and Nectars, Czech Agricultural and Food Inspection (CAFI), experts of the Central Customs Laboratories of the Ministry of Finance, Department of Food Preservation and Meat Technology of the Institute of Chemical Technology and other institutions have launched a campaign to improve the quality and authenticity of fruit juices in the Czech Republic. In the framework of this campaign tens of 100% orange juice samples were analysed every year in the period 1996–2001. The aim of the presented paper is to summarise the development of authenticity of orange juices in the Czech market in the last years.

MATERIAL AND METHODS

Samples of 100% orange juices: Hohes C, YO, Hello, Dolly, Fruity, Prim Jus, Džus, Oasa, Emig, Pomerančový juice, Don Simon, GOLD Glocken, Relax, Happy Hit, Koruna, Bari, Tesco, Libella, Pfanner, Happy Day, Donald Duck, Cappy, Fruiko, Best, Pomerančová šťáva, Vitajus pome-ranč, Aro – pomerančová šťáva, etc.

The analyses of main sample groups were carried out in the years 1996/97, 1998, 1999/2000 and 2001, each time the group of samples available in the market was analysed. Some of the products analysed in 1996 do not exist any longer, the product need not represent the same quality throughout this period. Analyses of smaller groups or individual samples of orange juices were made in all periods.

Preparation of samples: Five grams of orange juice were placed in a 100ml volumetric flask and the volume was made up to the mark with distilled water. The content of the flask was filtered through a filter with small pores and the filtrate was used for an analysis of sugars. For determination of cations and organic acids deionised water was used instead of distilled water and the filtrate was diluted with deionised water at a 1:10 ratio.

Analytical Procedures

Cations – Na, Ca, Mg, K were determined by capillary isotachophoresis with conductivity detection. 7.5mM H_2SO_4 + 7mM 18-crown-6 + 0.1% HPMC was used as a base electrolyte and 5mM BisTrisPropan + 10mM caproic acid as a terminating electrolyte. The initial and final applied field current 80 µA and 30 µA, respectively.

Sugars – Glucose, fructose and sucrose were individually quantified by HPLC on an Ostion analytical column (OSTION LG KS 0403 (4.2 % DVB), 6.3 µm, 250 × 4 mm) under the following conditions: mobile phase of degassed distilled water at a flow rate 0.5ml/min, temperature 80°C, pressure 8MPa, and refractive index detector. Samples were analysed by filtering through a 0.45 µm filter, injecting 20 µl, and measuring peak heights. An external standard method was used for the determination of particular sugar.

Organic acids (KVASNIČKA *et al.* 2002) **–** citric acid, isocitric acid, and malic acid were determined using capillary isotachophoresis with conductivity detection. The mobility of citric and isocitric acid is very close practically in the whole range of used pH, that is why the formation of a complex of citric acid with Ca^{2+} is used.

Formol number (VdF 1987*)* **–** titrimeric method according to RSK was used for the determination of formol number (raw data without correction to ammonia were processed).

Flavonoid glycosides (VdF 1987*)* **–** spectrometric analysis of flavonoid glycosides (hesperidin, naringin) at 420 nm according to Davis was used.

Refractive index – digital refractometer (Palette 100, Japan) was used for the determination of refractive index.

Relative density **–** relative density was determined by a gravimetric method at 20°C from differences between the weights of full and empty pycnometer.

Proline (EN 1141: Fruit and vegetable juices – Spectrometric determination of proline content) – proline was determined by a spectrophotometric method according to European standard.

Statistical Processing of Results

The data were processed by the statistical methods (linear regression, PCA, cluster analysis, etc.) included in the "Statistics for Windows" software.

RESULTS AND DISCUSSION

The results of analyses obtained throughout the years allow to evaluate the development of methods of adulteration of the commodity. While at the beginning of the project in 1996 more cases of "primitive" adulteration were discovered, recently more sophisticated and "technological" (pulp wash addition) adulteration has been found. In 1996 the most common methods of adulteration were: lower refractive index, significant lowering of fruit content, synthetic mixtures, undeclared addition of sugar and unsuitable water used for reconstitution. The general situation was better in 2000; more than 40% of samples complied with the AIJN. The most frequent examples of adulteration were: pulp wash addition ("no names", discount products), lower refractive index (including the imported juices), undeclared sugar addition and unsuitable water for reconstitution (higher Ca content). The development of juice authenticity is obvious from a comparison of the distribution of results in 1996 and 2000 for selected markers given in Figs. 1 and 2.

A higher content of calcium (according to the AIJN the range for calcium is 60–150 mg/l) is an indicator of higher pulp wash content in juice or higher pressure squeezing, another, often more important reason is the use of reconstitution water with higher content of calcium. The decision between these two causes depends on the values for other pulp wash indicators such as flavonoid content, polygalacturonic acid content and others. It is evident from the comparison in Fig. 1 that this parameter was the same or worse in 2000; it may be caused by a higher number of pulp wash discount low-end products in the market. The other marker in Fig. 1, the citric/isocitric acid ratio is an indicator of fruit content. Citric acid is usually added to mask the sugar addition, the higher the citric/isocitric acid ratio, the lower the fruit content. According to the AIJN this parameter should be lower than 130. It is obvious from the results in the figure that the samples analysed in 2000 fulfilled this criterion, the fruit content in juices of 2000 was higher.

Refractive index, the content of soluble solids is one of the principal AIJN parameters, according to the Code of

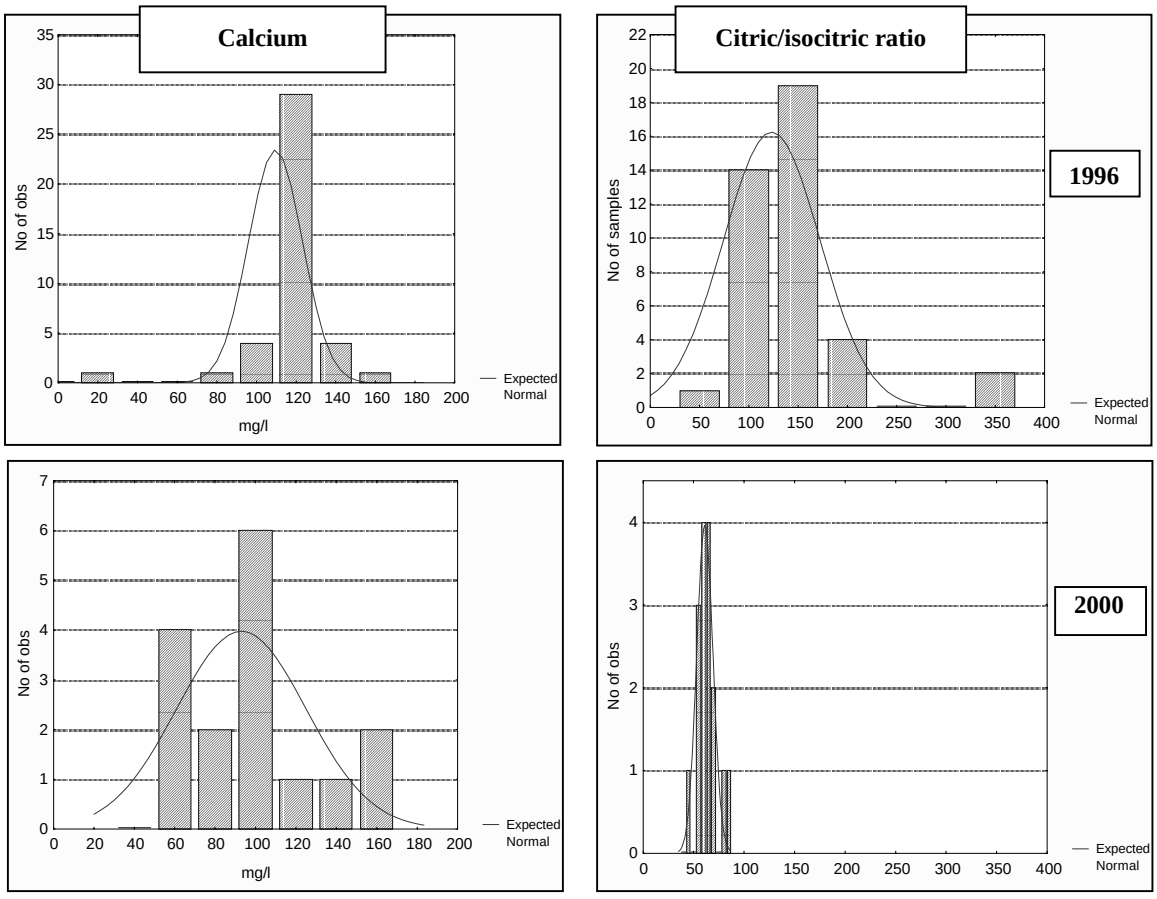


Fig. 1. Distribution of results for the markers calcium (AIJN range: 60–150 mg/l) and citric/isocitric acid ratio (AIJN: max. 130), comparison of the groups of samples analysed in 1996 and 2000 (Gaussian curve represents "expected normal" for the analysed samples, not the figures expected according to AIJN COP)

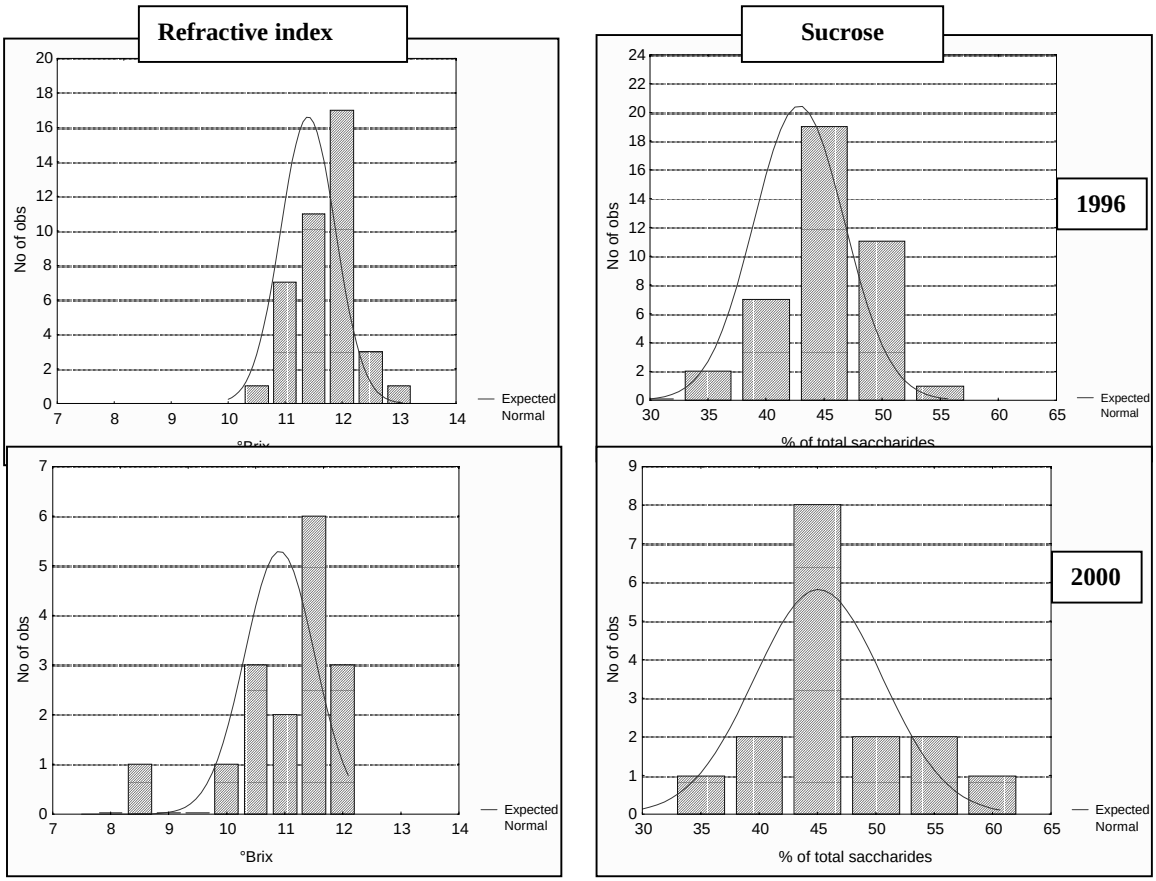
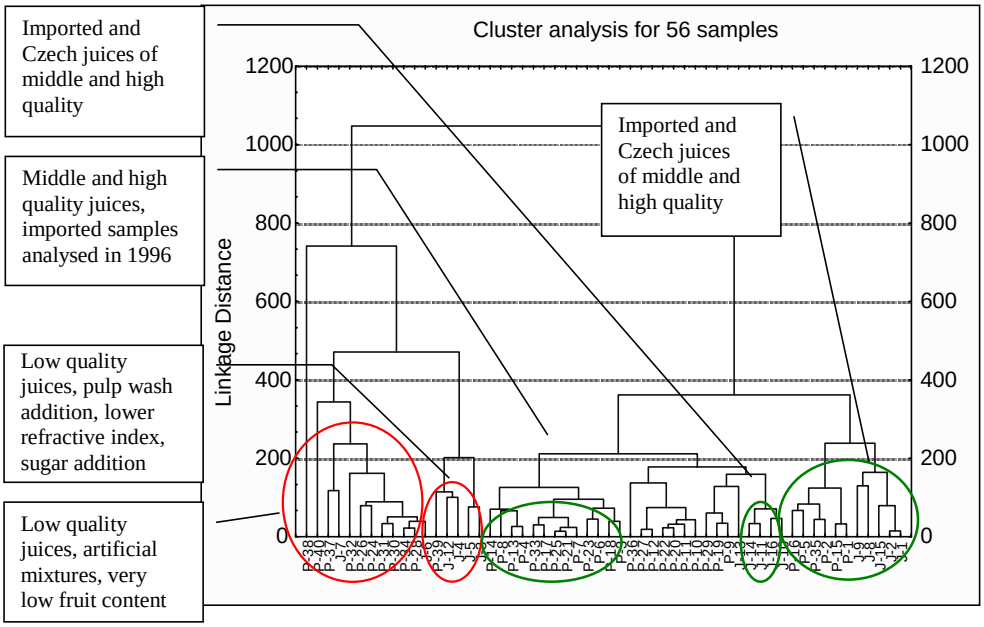


Fig. 2. Distribution of results for the markers refractive index (AIJN: min. 11.2°Brix) and sucrose (AIJN: sucrose – max. 50% of total mono- and oligosaccharides), comparison of the groups of samples analysed in 1996 and 2000 (Gaussian curve represents "expected normal" for the analysed samples, not the figures expected according to AIJN COP)



Samples P – 1996, samples J – 1999/2000

Fig. 3. Cluster analysis representing the groups of samples analysed in 1996 and 1999/2000

Table 1. Results of analyses of selected markers of two "100%" orange juice samples from the Czech market at the end of 2001

Parameter	Sample no. 1	Sample no. 2	Code of Practice AIJN
Refractive index (°Brix)	11.4	11.3	min 11.2
Sucrose (g/l)	73.3	62.7	10–50
Glucose (g/l)	15.9	22.7	20–50
Fructose (g/l)	16.3	22.7	20–50
Glucose:fructose	0.98	1	max. 1
L-malic acid (g/l)	0.8	0.6	0.8–3.0
Citric acid (g/l)	6.17	6.51	6.3–17.0
D-isocitric acid (mg/l)	60	42	65–200
Citric acid: D-isocitric acid	103	155	max. 130
Potassium (mg/l)	827	471	1300–2500
Formol index (ml 0.1 mol NaOH/100 ml)	13.9	6.9	15–26
Hesperidin – Davis (mg/l)	534	287	usually about 800 max. 1000

Practice the limit for 100% orange juice prepared by reconstitution from a concentrate is 11.2°Brix. The limit of the former Czech standard for fruit juices was 10°Brix. The recent legislation does not specify the limit; the refractive index is still the problem of juices in the Czech market. It is evident from the comparison of the results in 1996 and 2000 that the situation in this parameter is still bad. Moreover, the refractive index lower than 11.2 was also detected in several imported juices of EU producers. The relative content of sucrose (sucrose/sum of sucrose, glucose, fructose) is another simple marker that allows to detect the addition of sucrose. According to the Code of Practice the maximum relative content of sucrose for natural juices is 50%. It is obvious from the comparison of the results in 1996 and 2000 that the higher number is an improvement in this parameters, but there were several samples with added sucrose in 2000. This parameter is very easy to falsify, inverted sugar is added instead of sucrose, therefore the marker could be used to detect a primitive adulteration, in the case of more sophisticated ways of adulteration including the masking of adulteration the only procedure is an isotope analysis.

In the year 2001 the situation was similar like in 2000, but one producer started the production of juices with very low fruit content again. The examples of results of two orange juices sold under different names are given in Table 1.

The limited number of selected markers does not allow to evaluate the fruit content in juices; both samples have lower content of potassium, lower content of flavonoids and lower formol number. The fruit content in sample 1 could be about 60–70%, sample 2 is worse with estimated fruit content of about 30–40%. Sugar (sucrose) was added to both samples.

All data were processed by the cluster analysis (Fig. 3). The results are explained in the figure, but the most promising result seems to be the classification of selected products of Czech producers into the groups with high quality imported samples. On the other hand, some of the imported samples were comparable with the Czech samples of low quality.

A conclusion can be drawn that the quality of fruit juices will improve in future; the Association of Czech Producers of Fruit Juices and Nectars prepares a self-control system similar to that used in the EU countries. The Code of Practice has been implemented into national standards while the Czech standards are not obligatory but they will be used as a complement of Decree No. 335/1997 with latest amendments, to complete the specifications of juices e.g. in legal actions.

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Souhrn

VOLDŘICH M., SKÁLOVÁ P., KVASNIČKA F., CUHRA P., KUBÍK M., PYŠ P. (2002): **Autenticita 100% pomerančových šťáv na českém trhu v letech 1996–2001.** Czech J. Food Sci., **20**: 83–88**.**

V letech 1996 až 2002 byly analyzovány vzorky 100% pomerančových šťáv získané na tuzemském trhu. Kvalita a autenticita šťáv byla posuzována podle Code of Practice of AIJN (byly sledovány vybrané parametry – K^+, Na^+, Ca^{2+}, Mg^{2+}, obsah kyseliny citronové, isocitronové, jablečné, poměr citronová/isocitronová kyselina, obsah glukosy, fruktosy, sacharosy, sorbitolu, formolové číslo, obsah flavonoidů podle Davise a HPLC, obsah refraktometrické sušiny a další). V průběhu sledovaných let se zvyšovala kvalita a autenticita produktů. V roce 1996 byly hlavní případy falšování: a) snižování podílu ovoce, b) významné přídavky sacharosy maskované přídavky kyseliny citronové, c) několik případů "syntetické" pomerančové šťávy. V následujících letech se situace mírně zlepšovala, hlavní problémy zjištěné v letech 2000/2001 byly: a) nižší refraktometrická sušina, b) přídavky pulp wash, c) horší kvalita vody použitá k rekonstituci, d) nedeklarované přídavky sacharosy. Odchylky od požadavků AIJN nebyly zjištěny pouze v případě šťáv českých výrobců, ale také v několika případech diskontních a levnějších značek zahraničních výrobců vyrábějících pro český trh. Na základě výsledků jsou diskutovány možnosti zlepšení kvality a autenticity šťáv na tuzemském trhu (zavedení ČSN přejímající požadavky Code of Practice of AIJN, vytvoření systému samokontroly svazem výrobců, účast v evropském projektu European Quality Control System [EQCS] apod.).

Klíčová slova: pomerančová šťáva; autenticita; detekce falšování; ovocný podíl

Corresponding author:

Doc. Ing. MICHAL VOLDŘICH, CSc., Vysoká škola chemicko-technologická, Ústav konzervace potravin a technologie masa, Technická 5, 166 28 Praha 6, Česká republika

tel.: + 420 2 24 35 30 12, fax: + 420 2 33 33 37 73, e-mail: michal.voldrich@vscht.cz