Past Perfect, Present Perfect, Future Perfect—Quality Assessment and Quality Control Mechanisms at Beilstein†

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In the constantly expanding world of chemical information systems, the word "Beilstein" has always been regarded as synonymous with high quality, reliability, and comprehensiveness. In order to preserve these important criteria, a number of quality control mechanisms are applied at all production stages involved in the creation of the Beilstein data-pool from which all Beilstein products are derived. These mechanisms include the application of both manual (intellectual) data selection processes as well as a number of sophisticated automatic checking methods to each piece of data. Consequently, the quality and reliability of all Beilstein information tools is assured. This lecture surveys and details these quality control methods and includes some representative examples demonstrating the effects of the various assessment procedures.

1. INTRODUCTION: FROM INFORMATION TO INNOVATION

Nowadays more and more people, particularly those engaged in the field of science and technology, are beginning to appreciate the value of INFORMATION, and, due to its growing importance, it must be accorded equal consideration with other and more familiar production factors:

−raw materials
−real estate
−manpower
−machines
−capital
−energy
−INFORMATION

As an essential component of innovation leading to the development of new products, methods, and technologies, high quality factual information provides modern society with enormous advantages. It serves to avoid pitfalls and erroneous conclusions as well as preventing the age old problem of "reinventing the wheel". Instead it directs men, machines, investment, and energy in the right direction, that is, by concentrating effort on the important subjects in an evolutionary manner.

This is particularly true of *chemistry*, which is a scientific discipline with an enormous output of, and demand for, data.

At the moment, the growth of the chemical literature runs at about 500 000 publications per year, describing around 300 000 new (!) compounds.

Furthermore, since there is no indication that the increase in chemical information will either slow down or stop within the foreseeable future, we shall therefore have to cope with the growing flood of chemical information.

2. QUALITY ASSESSMENT AND QUALITY CONTROL MECHANISMS AT BEILSTEIN[1−5]

BEILSTEIN's answer to this problem has been twofold: *First*, creation of electronic counterparts to the well-renowned Beilstein Handbook, thus providing powerful tools to store and handle the avalanche of chemical information; *second*, perpetuation and perfection of what has always been Beilstein's "number one goal": *critical evaluation* of the published material.[1]

In this connection, "critical evaluation" ("scientific screening", "scientific scrutinizing") means the competent judgment of information with respect to its validity (i.e., its compatibility with the generally accepted state-of-the-art), its novelty, and its overall scientific importance.

BEILSTEIN, which is synonymous for an integrated, comprehensive, and quality-oriented multimedia information system, at present consists of the following main components:

−Beilstein's Handbook of Organic Chemistry
−Beilstein Database
 *Online (STN, DIALOG)
 *Inhouse (CrossFire)
−Beilstein Current Facts (CD-ROM)
−Beilstein Software
 *SANDRA
 *AUTONOM
 *MOLTERM

For more than 110 years, Beilstein has provided chemists with the world's most comprehensive collection of structures, properties, data, and associated information on organic compounds. All relevant data has been carefully collected and scientifically scrutinized, thus ensuring the highest quality of the information.

The editors not only point out errors in the published data but also check assertions of any speculative nature against subsequent findings.

The individual steps of this quality-improvement procedure will be outlined and illustrated with some typical examples.

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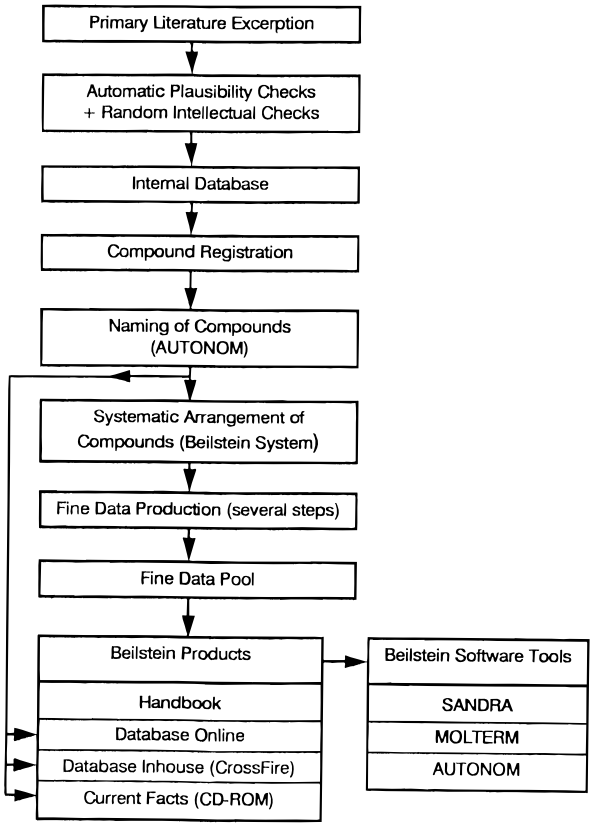
Table 1. Some Examples of Error Messages (Excerption)

no. of error message	text (indication)
11	non-numeric input in numeric field
13	value out of range
15	numeric field: more than three exponential digits
19	numeric field: sign of exponent must precede the first digit of exponent
23	numeric field: missing digits behind decimal point
96	molecular formula: illegal electronic charge
102	molecular formula: unknown characters or missing parentheses
131	CODEN: incomplete CODEN. Full length must be given
142	molecular formula: illegal use of roman numerals. Formulations such as "(I)" and "(III)" are not allowed to specify valence

Scheme 1. Beilstein Production Flow (Overview)



2.1. Beilstein Production Flow (Overview). Scheme 1 summarizes the production flow at Beilstein, feeding Handbook, Database online, Database inhouse (CrossFire), and Current Facts.

2.2. The Individual Beilstein Quality Assessment Steps. Let us now examine the individual production steps at Beilstein with respect to their potential for quality assessment and quality improvement.

2.2.1. Excerption. Primary literature excerption for Beilstein is carried out essentially by external subcontractors commissioned by the Institute, who guarantee the quality of all delivered electronic excerpts. The completeness of those excerpts and their quality are controlled by the Institute.

Error-checking programs have been built into the excerption program, and they are applied by the excerptor as well as by our internal scientific staff. Checking includes, e.g.

- to ascertain if the CODEN, year, and volume number of the excerpted journal are in agreement
- the correctness of the CAS registry numbers
- matching of molecular and structural formulas, i.e., of the molecular formula given in the original publication, and the one calculated by the program from the structural diagram. If mismatching occurs, then the input cannot continue before the necessary correction has been made.
- for properties involving ranges ("from .... to"): is the second value greater than the first?
- are all parameters given (e.g., "temp" for "heat capacity")? If not, the input mask cannot be left.

Table 2. Ranges of Physical Properties (Excerption)

property	range from	range to	unit
optical rotation	−3000	+3000	degree
bond moment	0	10	debye
boiling point	−170	+500	°C
melting point	−200	+500	°C
critical density	0.1	5	g/cm^3
dipole moment	0	50	debye
enthalpy of vaporization	8350	126000	J/mol
fluorescence maximum	150	900	nm
refractive index	1	2	
dynamic viscosity	0	100	Pa*s
vapor pressure	10^{-5}	10^4	Torr

Table 1 lists some of the possible 170 error messages which the input program may create and which the excerptor must consider for his correction work.

At Beilstein, various additional error-checking programs are applied, some of which check, e.g.

- inconsistent stereochemical details for a compound
- abnormal valencies (e.g., pentavalent carbon)
- suspicious starting material−product relationships
- inadmissible characters in the molecular formula
- derivative for characterization without properties
- false nuclei in the NMR masks

(the reported isotope is not present in the structure).

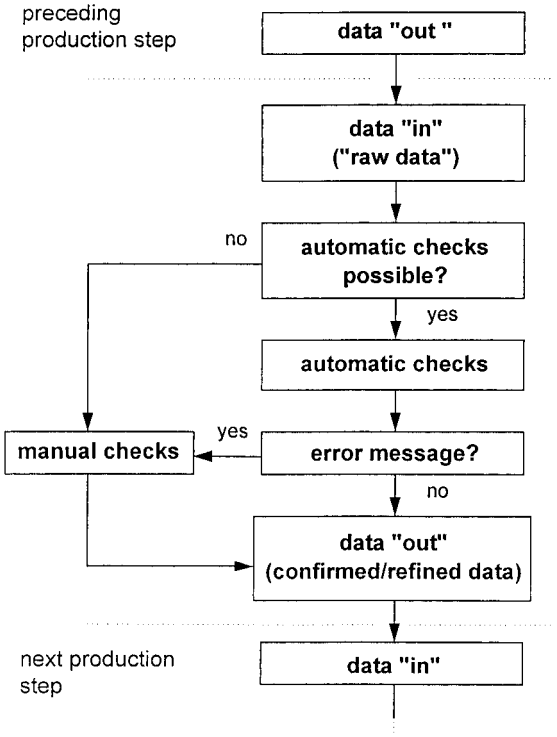
In addition, check programs also control the realistic limits of certain properties (see Table 2 for some examples). About 5−8% of all input from the excerptors is manually checked at random by our internal control staff, moreover the diskette material is directly compared with the relevant original literature. For all noted errors, the excerptor receives from his subcontractor an extensive list of errors with explanations, which acts as an aid for avoiding similar mistakes in the future.

All subcontractors are contractually obliged to maintain a minimum quality standard, i.e., no more than 60 error points per 100 compounds. If however, this limit is breached, then the entire monthly delivery is returned and must be improved without recompense. On the other hand, work of exceptional

Table 3. Error Point Catalogue (extract)

error	no. of error points
wrong constitution or configuration	20
wrong citation	20
missing compound	10
missing property	5
missing compound name	4
missing solvent, reaction condition	2
typing error	1

Scheme 2. Beilstein Fine-Data Production Steps



quality, i.e., fewer than 30 error points per 100 compounds, is rewarded with a bonus. The basis of the awarding of error points is a so called "Error Point Catalogue" which constitutes part of the contract between Beilstein and the subcontractor.

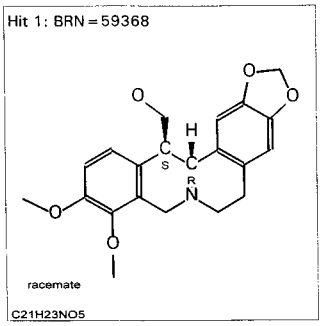
Table 3 shows an extract from this catalogue. For any one compound, the total number of error points must not exceed 20, and for any particular property the limit is 10 error points.

In our experience this system has worked extremely well. Most data, delivered by our subcontractors, is of the highest quality as a result of the above mentioned financial penalties and rewards.

After incorporation of the excerpted and—where necessary—corrected structures, data, and properties into our internal database, all compounds are registered. Names are then provided by our automatic naming program AUTONOM. All compounds with their associated properties and data are then fed into Current Facts, CrossFire, and the online database.

A computerized Beilstein Ordering Algorithm is then applied to all structures, thus determining unequivocally the place of each individual compound in the Beilstein Handbook.

It should be noted, however, that the systematization of structures according to this system is more than just a necessary means for arranging compounds in the Handbook.



Related Structure - RSTR

Note 1	Diese Konfiguration kommt wahrscheinlich der nachstehend beschriebene n Verbindung aufgrund ihrer Bildungsweise in Analogie zu (+)-Thalictricav in (S 6500) zu.
Ref. 1	Naruto; Kaneko, YKKZAJ, Yakugaku Zasshi, 92 <1972> 1017, 1018, Chem.Abstr.:77 <1972> 114605

Figure 1. Example of "scientific screening" (taken from CrossFire) (copyright 1995, Beilstein Institute).

Since the system brings chemically related compounds into close proximity, it is an indispensable prerequisite for the scientific data-evaluation which significantly contributes to the quality assessment for *all* components of the Beilstein Information System.

2.2.2. Fine-Data Production. Whilst by this stage numerous sophisticated automatic as well as manual checks have already been applied to the primary literature excerpts, and any irregularities have been corrected, the fine-data production steps described in the following are the core of the quality assessment at Beilstein. Here most of the additional nonautomatic "intellectual" (manual) checks are carried out by our scientific staff. This leads to the clarification of dubious results, the verification of reported data, the precise assignment of stereochemistry, and so on.

According to Scheme 2, a proven and well-examined combination of automatic and intellectual (manual) checks is applied to all of the following individual fine-data production steps. After completion of the critical evaluation and quality-improvement steps described in the following sections, the resulting improved data, constituting the Fine-Data Pool, will either complement or replace the initial data.

We just started a program to further speed up this quality-improvement process, ensuring that *all* Beilstein products will contain the same high-standard quality data at the same time.

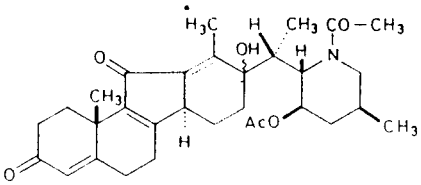
2.2.2.1. Automatic Checks. During the individual fine-data production steps, mentioned in Scheme 2, in principle the same programs and error-locating algorithms are used as mentioned earlier for the excerption stage.

Additional programs at the control stages also check

- —if the compounds named have been duplicated
- —if compounds ending "-ium" have a positive charge
- —if all sets of brackets in the compound name are paired
- —if the numbering corresponds to the actual number of substituents in the names (e.g., *2,4,6-trin*itrophenol)

Some examples of diagnostic messages, generated by the automatic error-checking programs applied, are

- —keyword "termolysis" unknown (correct: thermolysis)
- —reagent "H_2So_4" unknown (correct: H_2SO_4)



XV

(23*R*)-23-Acetoxy-28-acetyl-17-hydroxy-(17ξ*H*)-veratra-4,8,12-triene-3,11-dione $C_{31}H_{41}NO_6$, formula XV.

Configuration at the positions 22 and 23: inferred from the genetic relationship to isojervine (E III/IV **21** 6555).

Prep. From (23*R*)-28-acetyl-23-hydroxy-veratra-4,8,13(17)-triene-3,11-dione, by treatment with (i) $PhCO_3H$, (ii) Ac_2O [Py], and (iii) Al_2O_3 (*Wintersteiner, Moore*, J. Org. Chem. **29** [1964] 270, 275). – mp: 202–204.5° [from acetone + MeOH]; $[\alpha]_D^{21}$: +305° [$CHCl_3$]; IR; UV.

Figure 2. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 21/13, p 556) (copyright 1990, Beilstein Institute).

- –reagent "KO^{t}Bu, BuOH" unknown (correct: KOBu^t, Bu^{t}OH)
- –reagent "aq. H_2O" unknown (correct: aq. EtOH)
- –reagent "4-phenyl-but-3-en-2-one tosylhadrazone" unknown (correct: 4-phenyl-but-3-en-2-one (toluene-4-sulfonyl)hydrazone).
- –reagent "$LiAlH_4$*H_2O" unknown (correct: $LiAlH_4$; the original publication said: "lithium aluminum hydrate" instead of "~hydride")
- –caution: structure with doubtful stereo-descriptors
- –caution: compounds with exotic factual data mask(s)
- –caution: compound with uncertain source of C

Action resulting from these diagnostic messages is then taken in the following manual step within the fine-data production.

2.2.2.2. Manual (Intellectual) Checks. Although numerous automatic plausibility checks facilitate our quality assessment procedures at Beilstein, nevertheless the competent scientist is indispensable. It is his/her qualified evaluation which finally leads to the inclusion, correction, or—sometimes—even rejection of published data. This is especially true for so-called "multipublication compounds", where thousands of pieces of data must be evaluated.

Based on their scientific expertise, our chemists are, for example, able

- –to filter out multiple publications with the same principal content, well-known results, and trivial findings
- –to take into account corrections to earlier findings and to transfer them to similar cases
- –to recognize and, where possible, clarify conflicting data on a given subject
- –to correct incorrect structural assignments of compounds
- –to correlate individual results with analogous cases and reveal errors

Some of the criteria checked during the intellectual screening and data-evaluation procedure are summarized below:

- –material correctness of information (consistency with current general scientific principles)

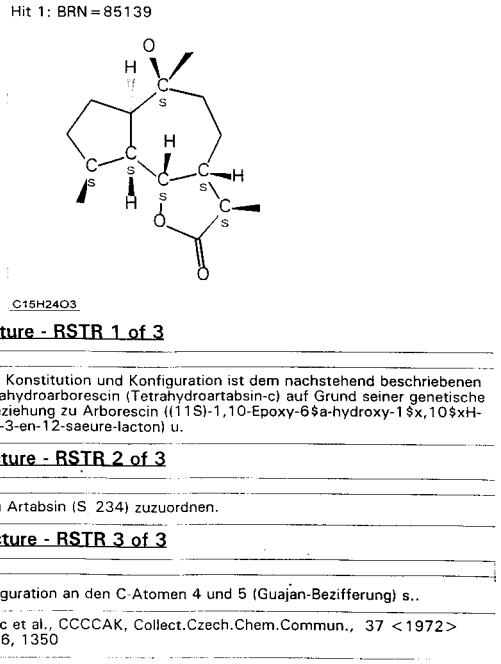


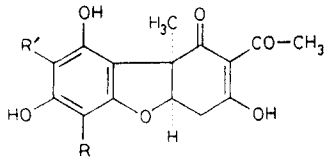
Figure 3. Example of "scientific screening" (taken from CrossFire) (copyright 1995, Beilstein Institute).

- –depth of information
- –completeness of information (are all relevant details and parameters given?)
- –objectives of the publication
- –accessibility of (primary) information (type of journal, language, date)
- –origin of the information

Some examples of the result of this intellectual screening are provided in Figures 1–3.

Stereochemistry. Assignments, corrections, or verifications of (doubtful) stereochemical features are areas which require the particular attention and expertise of the Beilstein Institute's editorial staff.

Uncertainties relating to the configuration of chemical compounds, detected in the primary literature, are cleared up by further literature research or by analogy reasoning.

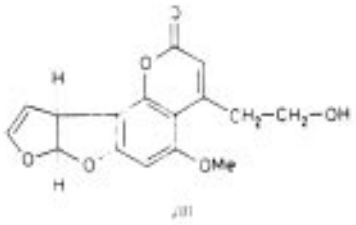


XIV

(4a*R*)-2,6-Diacetyl-3,7,9-trihydroxy-8,9b-dimethyl-(4a*r*,9b*c*)-4a,9b-dihydro-4*H*-dibenzofuran-1-one, (−)-Dihydrousnic acid $C_{18}H_{18}O_7$, formula XIV (R = CO-CH$_3$, R′ = CH$_3$) and taut. (E III/IV 3444).

Absolute configuration: inferred from the genetic relationship to (+)-usnic acid (p. 586). – ^{1}H-NMR (*Shoji*, Chem. Pharm. Bull. **10** [1962] 483, 485; *Shibata, Taguchi*, Tetrahedron Lett. **1967** 4867, 4870; *Kutney et al.*, Can. J. Chem. **54** [1976] 3721, 3722). IR [5000–

Figure 4. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 18/5, p 466) (copyright 1987, Beilstein Institute).



(7a*R*)-4-(2-Hydroxy-ethyl)-5-methoxy-(7a*r*,10a*c*)-7a,10a-dihydro-furo[3′,2′:4,5]furo[2,3-*h*]-chromen-2-one, Aflatoxin-B$_3$, Parasiticol $C_{16}H_{14}O_6$, formula VIII.

Configuration: inferred from the assumed biogenetic relationship to (−)-aflatoxin-B$_1$ (p. 566).

Isolation from cultures of Aspergillus flavus (*Heathcote, Dutton*, Tetrahedron **25** [1969] 1497, 1499; *Stubblefield et al.*, J. Agric. Food Chem. **18** [1970] 391) and cultures of Rhizopus species (*Cole, Kirksey*, J. Agric. Food Chem. **19** [1971] 222). – mp: 239–241° [from $CHCl_3$ + hexane];

Figure 5. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 19/10, p 544) (copyright 1988, Beilstein Institute).

Consequently, there are numerous stereochemical assignments found in Beilstein, which have not been given in the primary literature or elsewhere. This type of clarification is possible when one considers, for example

- genetic relationships between the compound with configurational uncertainty and compounds whose configurations have been established
- synthesis of the compound by methods analogous to those leading to compounds of known configuration
- degradative reactions, whose steric courses are known with certainty and which lead to the formation of the compound in question
- comparison of physical data within a homologous series

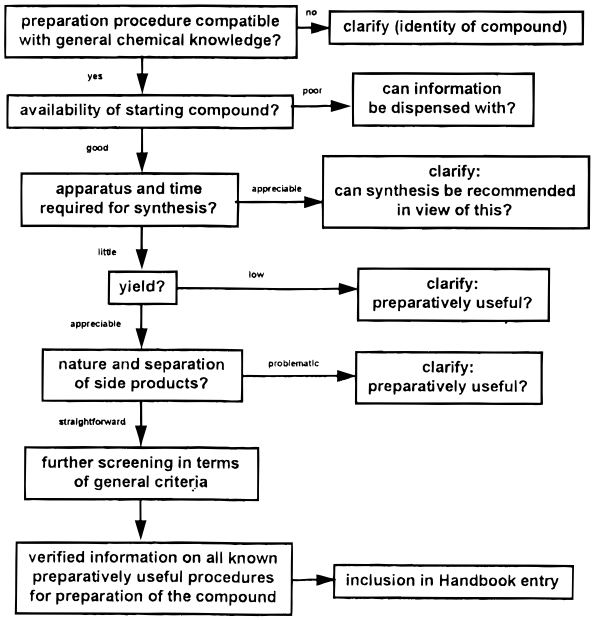
As a net result of our work at Beilstein, critical processing leads to a correction or verification of the constitution and/or configuration of ***almost every tenth compound reported in the primary literature, containing stereochemistry relevant structural features***.

Figures 4 and 5 show some representative examples.

2.2.3. Special Screening Procedures for the Handbook Entries. While the Beilstein database contains *all* information found in the primary literature, the publication of the printed handbook, for obvious reasons, must be restricted to a concise extract of reliable (best available) data.

Additional intellectual selection criteria are therefore used for the inclusion of data in the Handbook.

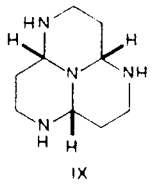
Scheme 3. Special Screening Procedures for the Handbook Entries on the *Preparation* of a Compound



The practical application of these additional intellectual selection criteria to processing of the Beilstein Handbook entries is exemplified by Scheme 3.

Similar screening procedures are applied to *all* types of data to be included in the Handbook (e.g., isolation from natural products; physical data; reaction data; and so on).

Figures 6–10, taken from the printed Handbook, illustrate the results of this additional quality assessment.



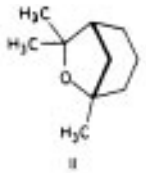
IX

(3ar,6ac,9ac)-Dodecahydro-1,4,7,9b-tetraaza-phenalene $C_9H_{18}N_4$, formula IX + mirror image.

Configuration: inferred from the analogous preparation of 2,5,8-trimethyl-(3ar,6ac,9ac)-dodecahydro-1,4,7,9b-tetraaza-phenalene stereoisomers (E III/IV **26** 1708; E V **26/11** 127).

Prep. From propenal and NH_3 [MeOH; 1250°; 100 at] (*J.L. Van Winkle et al.*, J. Org. Chem. **31** [1966] 3300, 3304). – Interatomic distances and angles [X-ray diffraction] of the trihydrochloride hemihydrate; density of the crystals: 1.2–1.4 (*A.E. Smith*, Acta Crystallogr. **19** [1965] 248, 253). mp: 118–120° [subl. sample]; $bp_{0.1}$: 117°; IR; pK'_{a1}, pK'_{a2} and pK'_{a3} [H_2O; potentiometrically determined]: 3.3, 5.8 and 7.6 (*Van Wi. et al.* 3301, 3304).

Figure 6. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 26/11, p 122) (copyright 1994, Beilstein Institute).



II

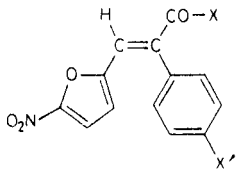
(1*R*,5*S*)-5,7,7-Trimethyl-6-oxa-bicyclo[3.2.1]octane, (1*S*,3*R*)-1,3-Epoxy-*m*-menthane, *m*-Cineole $C_{10}H_{18}O$, formula II (cf. E III/IV 213).

Configuration: inferred from the genetic relationship to (1*S*,3*R*)-*m*-menthane-1,8-diol (E IV **6** 5251).

bp_{20}: 76°; d_{20}^{20}: 0.9101; n_D^{20}: 1.4550; ^{1}H-NMR; IR (*Abraham, Verghese*, Indian J. Chem. **10** [1972] 140).

This configuration may also be inferred for a compound ("silvecineol"; bp_{733}: 180.8°; n_D^{20}: 1.4545; $[\alpha]_D^{20}$: −67.1°) obtained by *Bardyshev et al.* (Dokl. Chem. (Engl. Transl.) **202** [1972] 102; orig. 834) on treatment of (1*R*)-1,8-dichloro-*cis*-*m*-menthane (E IV **5** 150) with NaOAc.

Figure 7. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 17/1, p 272) (copyright 1984, Beilstein Institute).



XII XIII

(*E*)-3-(5-Nitro-furan-2-yl)-2-phenyl-acrylic acid $C_{13}H_9NO_5$, formula XII (X = OH, X′ = H).

Configuration: inferred from the analogous preparation of (*E*)-3-furan-2-yl-2-phenyl-acrylic acid (p. 558).

Prep. From 5-nitro-furan-2-carbaldehyde and potassium phenylacetate [Ac_2O] (*Saikachi, Tanaka*, Yakugaku Zasshi **80** [1960] 1584, 1586; CA **1961** 7380; *Hirao, Kitamura*, Nippon Kagaku Zasshi **85** [1964] 506; CA **62** [1965] 11756). – Light-yellow cryst.; mp: 160–161° [from benzene] (*Sa., Ta.* 1586), 158.5–159.5° [from benzene] (*Hi., Ki.*). UV/VIS (*Yoshina, Tanaka*, Yakugaku Zasshi **88** [1968] 410, 411, 414; CA **69** [1968] 72454).

~Ethyl ester $C_{15}H_{13}NO_5$. Yellow cryst.; mp: 39–40° [from aq. EtOH]; UV (*Saikachi, Tanaka*, Yakugaku Zasshi **83** [1963] 147, 150, 152; CA **59** [1963] 3858).

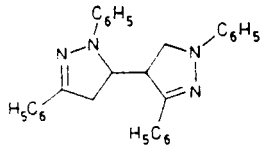
~Hydrazide $C_{13}H_{11}N_3O_4$. *Prep.* From the acid chloride (see below) and $N_2H_4 \cdot H_2O$ [Et_2O] (*Kato, Hirao*, Nippon Kagaku Zasshi **87** [1966] 1336, 1337; CA **67** [1967] 82172). – Orange-red cryst.; mp: 169–173° [from benzene].

Figure 8. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 18/6, p 559) (copyright 1987, Beilstein Institute).

3. CONCLUSIONS AND PROSPECTS

The advantages and benefits for the user of this kind of data-processing and the resulting quality improvement of chemical information, as outlined in this talk, are obvious:

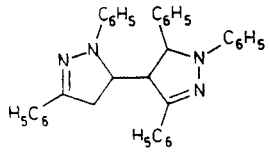
- improved confidence in the accuracy of the sifted data
- a greatly reduced risk of false conclusions and non-optimal experimental planning as a result of inexact literature results



III

2,5,1′,3′-Tetraphenyl-3,4,4′,5′-tetrahydro-2*H*,1′*H*-[3,4′]bipyrazolyl $C_{30}H_{26}N_4$, formula III.
The compound to which this structure was assigned by *E.E. Baroni, K.A. Kovyrzina* (J. Gen. Chem. USSR (Engl. Transl.) **33** [1963] 947, 949; orig. 959), should be reformulated as 3,2′,5′-triphenyl-4,5,3′,4′-tetrahydro-1*H*,2′*H*-1,3′-*p*-phenylene-bis-pyrazole (inferred after genetic relationship to 4-(3-phenyl-4,5-dihydro-pyrazol-1-yl)-benzaldehyde [E V **23/6** 401]).

Figure 9. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 26/12, p 35) (copyright 1994, Beilstein Institute).



XI

2,5,1′,3′,5′-Pentaphenyl-3,4,4′,5′-tetrahydro-2*H*,1′*H*-[3,4′]bipyrazolyl $C_{36}H_{30}N_4$, formula XI.
The compound to which this structure was assigned by *E.E. Baroni, K.A. Kovyrzina* (J. Gen. Chem. USSR (Engl. Transl.) **33** [1963] 947; orig. 959), should be reformulated as 3,5,2′,5′-tetraphenyl-4,5,3′,4′-tetrahydro-1*H*,2′*H*-1,3′-*p*-phenylene-bis-pyrazole (inferred from the genetic relationship to 3-[4-(3,5-diphenyl-4,5-dihydro-pyrazol-1-yl)-phenyl]-1-phenyl-propenone [E V **23/9** 20] and 4-(3,5-diphenyl-4,5-dihydro-pyrazol-1-yl)-benzaldehyde [E V **23/9** 19]).

Figure 10. Example of "scientific screening" (taken from Beilstein Handbook, EV, Vol. 26/12, p 180) (copyright 1994, Beilstein Institute).

- −intensive intellectual "re-examination" of selected data unnecessary
- −greater time saving
- −promotion of further innovations due to the cross-referencing of published information

It should be evident that the data quality assessment of primary information by means of a science-based, and intelligently carried out, evaluation process requires very high personnel and financial commitments. Despite these costs, we at Beilstein will strive to maintain our reputation of publishing only quality data,—in order to also justify the term "*Future Perfect*" in the title of this Award Address.

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